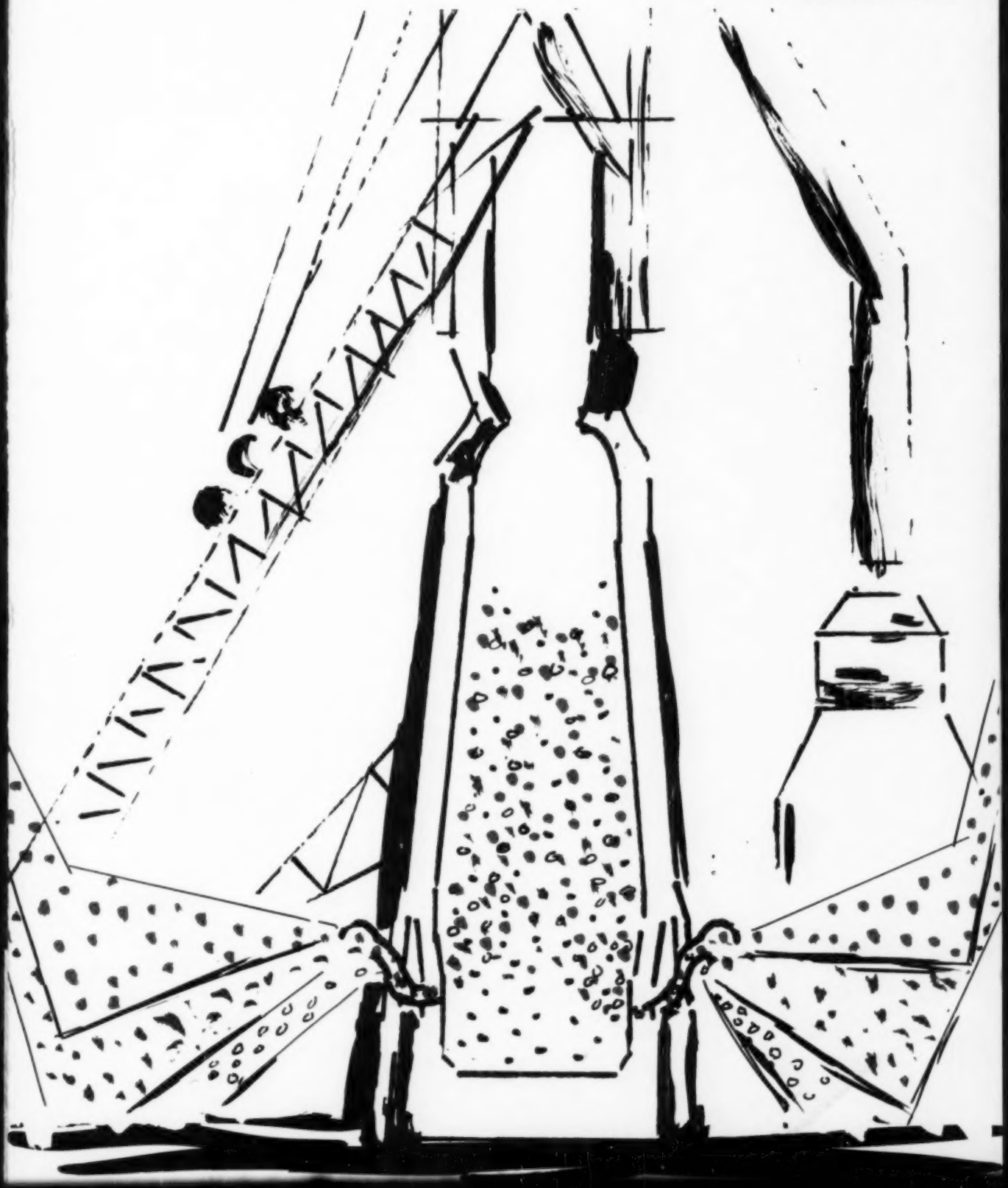
JOURNAL OF

METALS

JANUARY 1961



One of a series.

Why we eliminated the earth's magnetic field . . . almost

In an isolated laboratory in southwestern Ohio, GM Research scientists have reduced the earth's magnetic field to one ten-thousandth of its usual strength. This is about as weak as the interplanetary field detected by the Pioneer V solar satellite.

Why neutralize the earth's field? To perform with precision one of the more fundamental experiments in magnetism — measuring the Einstein-DeHaas effect. The measurement is simple in concept, experimentally difficult because of the tiny forces involved. It is made by suspending a ferromagnetic rod in a nearly field-free environment . . . magnetizing the rod . . . then measuring the effect (how much the rod rotates) when this known magnetization is reversed.

The beauty of the experiment is that the resulting values can be related directly to the motions of electrons in the rod. The values indicate the large portion of magnetization due to the *spin* of electrons . . . and the slight, but theoretically important, remaining portion due to *orbital* motion of electrons.

These measured values are helping scientists form a better understanding of the perplexing phenomenon — ferromagnetism. Currently being pursued in cooperation with the Charles F. Kettering Foundation, this long-standing project is one of the ventures in basic research of the General Motors Research Laboratories.

General Motors Research Laboratories

Warren, Michigan

Gyromagnetic Ratios

	a	***b***
Iron	*1.92*	*1.90*
Cobalt	*1.85*	*1.83*
Nickel	*1.84*	*1.83*
Supermalloy	*1.91*	*1.91*

Comparison of (a) gyromagnetic ratios measured in the new Kettering Magnetics Laboratory with (b) corresponding ferromagnetic resonance measurements. These ratios would equal 2 if magnetization were due only to electron spin, or 1 if due only to orbital electron motion.

System of Helmholtz coils used to neutralize earth's magnetic field.

JOURNAL OF

METALS

Vol. 13, No. 1
January, 1961

COVER: "Blast Furnace Injections—Key to Increased Productivity and the Future of Ironmaking?" might be an expression of the theme of this month's issue. It is depicted on the cover by David Cain and discussed in detail in a number of articles.

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EDITORIAL STAFF: Editor in Chief, F. Weston Starratt; **Associate Editor,** Edward Fitzgerald, **Production Editor,** Otto Johnson; **News Editor,** William Leggitt; **Editorial Assistant,** C. M. Cardon.
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personnel

● These items are listings of the Engineering Societies Personnel Service, Inc. This Service, which cooperates with the national societies of Civil, Chemical, Electrical, Mechanical and Mining, Metallurgical, and Petroleum Engineers, is available to all engineers, members or non-members, and is run on a nonprofit basis.

If you are interested in any of these listings, and are not registered, you may apply by letter or resume and mail to the office nearest your place of residence, with the understanding that should you secure a position as a result of these listings you will pay the regular placement fee. Upon receipt of your application a copy of our placement fee agreement, which you agree to sign and return immediately, will be mailed to you by our office. In sending applications be sure to list the key and job number.

● When making application for a position include eight cents in stamps for forwarding application.

—MEN AVAILABLE—

Metallurgist, BS, ChE (1937). Twenty-three years process development chem-met to the steel, foundry and electrical apparatus industries. Experienced in market development, technical sales, and production. Prefer East. M-247.

—POSITIONS OPEN—

Mill Representatives, 2, technical degree and/or background desired, with minimum of 3 years selling to O.E.M.s; prefer experience to be in the nonferrous metals field. $7000-$9000 year plus incentives; car expenses. Excellent opportunities. Headquarters New Jersey, but will be required to relocate. W9870.

Metallurgist or Chemist with at least an MS degree, for nonferrous laboratory research in minerals purification, induction melting and alloying. $7800-$8000 year. Long Island, N. Y. W9867.

Steel Process Mathematical Analysts, technical degree with emphasis on metallurgy or mathematics. Minimum of 3 years experience in steel or other metal processing industries. Must have background capability of relating various steel making processes in mathematical expressions. Pennsylvania. W9857.

Engineers. a) Two Production Engineers, preferably mechanical, experienced in metal working machinery, rolling, extrusion, slitting, wire drawing. Experience in soldering or jewelry company desirable. 1) To supervise night shift. 2) To supervise day shift (heavy experience in wire drawing). b) **Metallurgist,** graduate, experienced in precious metals or low melting soft alloys. New York Metropolitan area. W9852.

Junior Metallurgist, recent graduate, with one to two years experience, to assist chief metallurgist in selection of materials and heat treatment for specific applications. Quality control of incoming material. Company is manufacturer of paper mill machinery and machine knives. $5000-$7000 year. New England. W9850.

Research Metallurgist, PhD in physical metallurgy preferred; some background in solid state physics and chemistry desirable. Work will involve research and development on metal cutting tool materials, surface treatments, friction and wear studies. Salary open. Midwest. W9833.

Assistant Metallurgist or Chemical Engineer, graduate, with experience in metal processing, including electro-plating and finishing of materials. Military service completed. $6000-$8000. Morris County, N. J. W9824.

Research Assistant, BS in physical metallurgy, physical chemistry or physics. Opportunity for graduate study and fundamental research at major university leading to MS or PhD in materials science. Full time research during summers. New York State. W9791.

Fellowships and Research Appointments in metallurgy. Full time experimental and course work toward MS or PhD degree. BS degree with no experience, salary approximately $3600. Available July or September 1961. Pennsylvania. W9817.

AIME and Met. Soc. Publications Prices

1961 JOURNALS

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(One Journal With Dues)

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Journal of Petroleum Tech.	4.00	8.00
Journal of Metals	4.00	8.00

NONMEMBER SUBSCRIPTIONS

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Journal of Metals	$10.00	$12.00

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TRANSACTIONS VOLUMES

		Members		Nonmembers	
No.	**Society**	**Pay with Dues**	**Pay Later**	**North, South & Central America**	**Elsewhere**
217	Soc. of Mining Eng.	$8.50	$8.50	$12.00	$12.00
218	The Metallurgical Soc.	5.00	6.50	35.00	35.00
219	Soc. of Petr. Eng.	3.50	4.90	7.00	7.00

Note: 1960 bound TRANSACTIONS volumes to be issued in 1961.

Engineering Societies Library

This year's large increase in the use of the Engineering Societies Library, reported by its director Ralph H. Phelps, indicates a continually growing awareness of the Library's value to the engineering profession. Orders received by mail from individuals and companies all over the world, for services such as literature searching, translating, and photoprint and microfilm copying, increased 25 pct over last year. Requests received by telephone increased over 35 pct. Such non-visitor use now is 68 pct of total library use.

The Library has added 9 pct more volumes this year than last year. It receives, by subscription, gift, or exchange, 3400 periodicals and other serial publications—which are so important in keeping engineers abreast of the most current developments in their fields.

The Cataloguing Department catalogued more books and added more cards to the card catalog than in any one previous year. The Library Staff reviewed 691 books, providing book reviews for several journals, including those of Founder Societies. Staff members have also devoted much time to planning for the new Library facilities in the United Engineering Center, which will be occupied by the Library and some two dozen other engineering organizations in 1961.

Several books in the Engineering Societies Monograph series, sponsored by the Library and the Founder Societies, are being reprinted, and a new edition of a book in the series *Theory of Plates and Shells* by Stephen P. Timoshenko and S. Woinowki-Krieger was published. Arrangements were made so that Xeroxed reproductions of out-of-print monographs are now available through University Microfilms.

The Library has made outstanding contributions in the library field as well as in the field of engineering. *Alternatives to the Scientific periodical; A Report and Bibliography* by Ralph H. Phelps and John P. Herling appeared in UNESCO Bulletin for Libraries, v. 14, March-April, 1960, p. 61-75.

At the Houston works, Sheffield Division, Armco Steel Corporation, two 20-foot Lectromelt furnaces contribute high availability, accurate control and fast turn-around to volume production of high quality steels. For recommendations on furnace equipment for melting or smelting, get in touch with Lectromelt, world's largest manufacturer of electric furnaces exclusively. Lectromelt Furnace Division, McGraw-Edison Company, 326 32nd Street, Pittsburgh 30, Pennsylvania.

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Physicists,
Electro-Chemists . . .

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* * *

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- **Microstructure studies** of solid state by X-ray diffraction, fluorescence, electron microscope, electron microprobe.
- **Electrochemical work** of both practical and theoretical nature with bench scale and pilot operations to develop new plating and coating techniques.
- **Research on the effects of alloying elements** on ferrous and nonferrous casting alloys to develop new alloys and improve melting and casting.
- **Welding research** to develop new rods and techniques for existing and new alloys.

Requirements: Ph.D. or Sc.D. Also M.S. degree with research experience is acceptable.

More information is given in the current report of INCO activities, "Metals Plus Research." Do you want to review a copy?

Director of Research
The
International Nickel Company, Inc.
67 Wall Street, New York 5, N. Y.

personals

Richard N. Ames has been appointed technical services specialist for Kaiser Refractories & Chemicals div. in the Chicago area. He was formerly with U.S. Steel Corp.

Arthur K. Blough is now chairman of the open hearth and electric furnace committees of Republic Steel Corp., of Cleveland, Ohio.

Robert B. Brackin has been appointed manager of Columbium properties for Kennecott Copper Corp. He will be responsible for the company's columbium subsidiaries in Nigeria and Quebec. Before joining Kennecott, Mr. Brackin was with E. I. duPont de Nemours & Co.

William L. Bruckart has entered private practice as an industrial and marketing consultant in Philadelphia. He was formerly with Universal-Cyclops Steel Corp.

W. E. Boyd has been named manager of the Montreal, Que., Canada, office of Charles Bruning Co., Inc.

Richard S. Cremisio is now a Fellow of the Mellon Institute in Pittsburgh. He will be engaged in research on the effect of processing variables on metal properties. He was formerly staff metallurgist with Crucible Steel Co., of America.

Maurice F. Dufour, vice president of Freeport Sulphur Co., has been appointed director of development and research for the company.

Fred M. Gilles, chairman of the executive committee of Acme Steel Co., reached normal retirement age with the company on November 1. However, he has agreed to remain as senior consultant for steel operations, and as a member of the board of directors.

John J. Gilman is now professor of engineering at Brown University, Providence, R. I. He was formerly a metallurgist at the General Electric Research Laboratory, Schenectady, N. Y.

Robert B. Gordon has been appointed manager, technical operations of Atomics International, a subsidiary of North American Aviation, Inc., Canoga Park, Calif.

Edward M. Grala has joined the metallurgy div. of the Brush Beryllium Co. He was previously with the Lockheed Missiles and Space div., Sunnyvale, Calif.

B. J. Haley has been appointed manager of the Ashtabula Works of Union Carbide Metals Co. He has been assistant manager at Ashtabula since October 1956.

John H. Hoke has been appointed assistant professor in the Dept. of Metallurgy at Pennsylvania State University.

S. L. Hoyt has returned from a sixteen month mission to Europe for the Dept. of Defense. While abroad, he served in a liaison capacity to investigators in metallurgical and welding research. He will take up permanent residence in Berkeley, Calif., and resume private practice as a metallurgical consultant.

W. A. Krivsky has been appointed head of Brush Beryllium Co.'s newly-established Works Process Engineering Dept., at its Elmore, Ohio plant. **K. G. Wikle** has been named technical manager of beryllium alloy operations and chief metallurgist.

Robert W. Lindsay has been appointed head of the Dept. of Metallurgy at Pennsylvania State University. He was formerly professor of metallurgy.

William G. Pfann, metal research specialist with Bell Telephone Laboratories, has been awarded the 1960 Professional Progress Award of the American Institute of Chemical Engineers.

J. Donald Rollins will become president of the American Bridge div., of U.S. Steel Corp., effective January 1. He is presently vice president, facility planning and appropriations.

Ralph A. Schaefer has been appointed director of research of the Electric

(*Continued on page 59*)

IMMEDIATE OPPORTUNITY FOR
PHYSICAL METALLURGIST

Large metropolitan New Jersey research laboratory currently engaged in semi-conductor and thermoelectric field seeks senior man with broad and proven background in physical metallurgy. Minimum of 5 years' experience; PhD desirable, but not essential. Salary open. Send complete resume to Box No. 17JM.

Excellent opportunity for B.S./M.S. Metallurgist. Minimum five years' experience; to establish and operate metallographic laboratory in large metropolitan New Jersey research laboratory engaged in semi-conductor and thermoelectric work. Must possess good laboratory technique as well as a sound theoretical background. Salary commensurate with experience. Send resume to Box No. 18JM.

METALGRAMS

UNION CARBIDE METALS

. . . news of "Electromet" ferroalloys and metals

JANUARY, 1961

CLEANER STEELS-HIGHER QUALITY -- "Silicomanganese deoxidation in the furnace produced average cleanliness better than any other furnace practices tried," the 1957 Open Hearth Conference was told. The cleaner steel produced with silicomanganese additions has better surface quality and mechanical properties. Silicomanganese not only produces fewer inclusions, but also forms more desirable inclusion types. Other advantages: improved forging and rolling properties, lower conditioning costs, and increased mill yield.

* * *

DATA FAVOR SILICOMANGANESE -- Production data has shown that lowest scarfing costs are obtained with a heavy silicomanganese block. Silicomanganese also reduces hot shortness of some high-sulfur steels and generally improves the quality of other steels. Long internal seams that cause rejections in the finished product are also reduced with a silicomanganese block. Have your Union Carbide Metals representative show you how you can improve the quality of killed steels used for all applications. Also, ask for the article, "Silicomanganese Produces Cleaner Steels", in the Winter 1961 issue of UNION CARBIDE METALS REVIEW.

* * *

LOW COSTS WITH CHARGE CHROME -- In recent years, the use of charge chromes in stainless steel melting has increased markedly. The reason: they are the lowest-cost ferrochromes available. Union Carbide Metals offers three alloys with different chromium, carbon, and silicon contents to meet individual practices. UCM's studies using a high-speed computer have helped steelmakers select the charge chrome that will give lowest costs. Look to Union Carbide Metals for help on the most economical use of charge chrome alloys. For more information, write for the article, "Predicting Minimum Materials Cost for Stainless Steel", in the Spring 1959 issue of UNION CARBIDE METALS REVIEW.

* * *

WHY BUY AMERICAN? -- Ferroalloy imports doubled between 1955 and 1959. Steel, iron, and nonferrous imports are up sharply too! Foreign producers have low wage rates which enable them to compete on a price basis with the U. S. However, they cannot compete with U. S. producers on three counts: (1) valuable metallurgical assistance at a user's plant, (2) consistent quality in all materials, and (3) products delivered when the customer wants them. For further information, write for the article, "Why Buy American?", in the Fall 1960 issue of UNION CARBIDE METALS REVIEW.

* * *

MANGANESE IN ALUMINUM -- In binary alloys, manganese improves the strength of aluminum by 20 per cent with no sacrifice in corrosion resistance. Thus, 3000 series alloys (containing 1.25 per cent manganese) are good general-purpose alloys for storage tanks, piping, furniture, TV antennas, awnings and siding, and chemical equipment. Manganese is also added to 2000, 5000, and 6000 series alloys to improve corrosion resistance. UCM offers low-iron ferromanganese and "Elmang" electrolytic manganese for such additions. More information can be obtained by writing for the article, "Manganese Puts Muscle in Aluminum", in the Winter 1961 issue of UNION CARBIDE METALS REVIEW.

* * *

UNION CARBIDE METALS COMPANY, Division of Union Carbide Corporation, 270 Park Avenue, New York 17, N. Y. In Canada: Union Carbide Canada Ltd., Toronto.

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new books

Books that are marked (•) may be ordered through AIME. Address Irene K. Sharp, AIME Book Dept., 29 W. 39 St., New York 18, N. Y. A discount is given whenever it is possible.

Book Reviews

In order to give more complete coverage to books dealing with metallurgy, the JOURNAL OF METALS will publish a short review of each important addition to metallurgical literature. These reviews will be written by well-known authorities in the particular field of metallurgy under consideration.

Contributions to the Data on Theoretical Metallurgy. XIII. High Temperature Heat-Content, Heat-Capacity and Entropy Data for the Elements and Inorganic Compounds by K. K. Kelley, U. S. Bureau of Mines, Bulletin 584, U. S. Government Printing Office, 232 pp. $1.25 (paper cover), 1960. *Reviewed by H. H. Kellogg of Columbia University.*

We have come to expect much for our money in the Bureau of Mines publications of K. K. Kelley, and this latest contribution is no exception. Nowhere else in the literature of chemistry or metallurgy can one find compilations of thermochemical data which rival those of Kelley for accuracy, completeness of coverage, and usefulness. Nor are these compilations simply uncritical collections of every reported measurement. Rather, for each substance considered, all existent measurements have been evaluated and compared and *best* values chosen with the critical acumen that we associate with Kelley's name.

This new volume is a revision of Kelley's earlier compilation (Bulletin 476, 1949) of high temperature heat-content data. Users will be glad to hear that the new bulletin includes data on 893 elements and compounds—about twice the number listed in Bulletin 476. The format of Bulletin 476 has been retained: values of $H_T—H_{298.15}$ and $S_T—S_{298.15}$ are tabulated for intervals of 100°C and empirical equations for $H_T—H_{298.15}$ and C_p versus temperature are given. Duplication of the data in units of calories—gm mole—°C and Btu-lb mole-°F, included in the earlier publication, has been dropped, and only the calories—gm—mole—°C units are used in the new bulletin.

Only two minor shortcomings occur to this reviewer. Although the accuracy of the empirical equations in reproducing the tabulated data is reported, there is no indication of the probable absolute accuracy for each set of data. The second objection is purely mechanical—without using the index it is difficult to locate data for a compound because an alphabetical key at the top of the page (such as any dictionary possesses) is lacking.

In this bulletin we have tangible evidence of where some very small part of our tax money goes, and it is hard to imagine a more useful result. No one concerned with thermochemical calculations will lack a copy of this bulletin on his desk, and it will be mentioned in the references to scientific articles from now until the next edition appears in 1970.

Feuerfestkunde, by Friedrich Harders and Sigismund Kienow, Springer-Verlag, Berlin, 981 pp., about $30, 1960—This extensive treatise on refractories is divided into sections by the major types; silica, fireclay, alumina, basic, and neutral, etc. Within each section are given the basic physical, chemical, and mineralogical characteristics, raw materials and their origin, manufacture, properties of the fabricated materials, and applications. Certain other insulating materials, such as asbestos and vermiculite, are also briefly

(Continued on page 12)

PHYSICAL METALLURGISTS

Outstanding opportunities are available for experienced research metallurgists; M.S. to Ph.D., to work on challenging, diversified research projects in such areas as:

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A two-pass blast furnace stove with application of TAMUL brick and shapes shown in red.

Increasing hot blast temperatures?

REINFORCE CRITICAL STOVE AREAS WITH TAMUL BRICK AND SHAPES!

Major blast furnace operators planning on increasing hot blast temperatures (to as high as 2000° F.) are now constructing stoves with dome, upper checkers, upper sidewalls, and combustion well walls of TAMUL (Taylor synthetic mullite) brick and shapes.

Excellent resistance to thermal spalling, alkali attack, and subsidence under long-term hot load, along with high refractoriness (P. C. E. — Cone 39) combine to make TAMUL the ideal refractory for these trouble spots. TAMUL should be laid up in new, improved TASIL cement.

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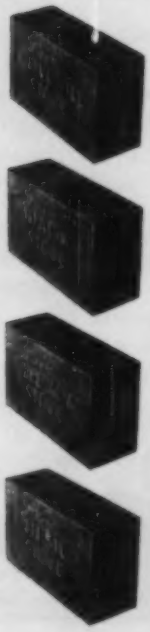

BRANDS FOR THE HOT BLAST STOVE

OLIVE HILL STOVE—Like OLIVE HILL BF for Blast Furnaces, OLIVE HILL STOVE is a highly accredited brand of high duty fireclay brick for hot-blast stoves, well known throughout the world.

HIAC-K STOVE—This superduty fireclay brick is widely used in well walls, domes and top checkers of hot-blast stoves that must endure severe operating conditions.

SUPERAC-K STOVE—High-fired superduty fireclay brick with their greater strength and volume stability are needed for the hotter sections of many stoves in high temperature operation. SUPERAC-K STOVE are outstanding in meeting this need.

HI-SIL STOVE—The most recent addition to the galaxy of GREFCO brands describes a semi-silica brick with extreme hot-load strength and spalling resistance. This brick is ideal for many hot-blast stove lining and checker applications.

...in brick

The most imperishable records in brick have survived *6000* years. The brick illustrated, for example, was used for making records at the Ur of the Chaldees, the City of Abraham.

The next best record in brick has been made by GREFCO linings. These brick were used for making *production* records.

GREFCO is proud of the many production records made on its Blast Furnace and Hot Blast Stove linings. GREFCO has specialized in the production of high quality brick for blast furnace linings and for hot blast stove walls, domes, rectangular checkers and multihole checkers. GREFCO's production facilities have been greatly increased and kept thoroughly modern in step with new technological developments.

GENERAL REFRACTORIES COMPANY

1520 Locust Street, Philadelphia, Pa.

BRANDS FOR THE BLAST FURNACE

OLIVE HILL BF—This brick is known throughout the world as an exceptional high duty fireclay brick for blast furnace linings.

OLIVE HILL HI FIRED—This blast furnace brick is made from the same materials as OLIVE HILL BF but is fired to higher temperatures to provide greater density, strength and better resistance to carbon disintegration.

SUPERAC-K-BF—This blast furnace brick is made from more superduty quality refractory clays, hard fired to develop increased density, resistance to spalling, and resistance to carbon disintegration. These brick can be used in zoned linings in combination with other blast furnace brands or can be used throughout the lining.

SUPERAC-K-BF (SR)—is a variety of SUPERAC-K-BF, that is formulated and processed to provide maximum slag and abrasion resistance. With these properties it is particularly suited for bottom blocks and those top areas of the furnace which are exposed to severe abrasion by the furnace charge.

SUPERAC-K-BF (NS)—another variety of SUPERAC-K-BF blast furnace brick, are expressly manufactured for increased spalling resistance, for lower inwalls where this property is sometimes of benefit.

education

Stanford Univ.

Organizational changes at Stanford University have added two new departments to the School of Engineering. The new Department of Materials Science, which was formerly the Metallurgical Engineering Department in the School of Mineral Sciences, is headed by O. Cutler Shepard, professor of mining and metallurgy. The Department of Chemical Engineering, formerly a div. of the Chemistry Department, is headed by David M. Mason.

NSF Grant For Brooklyn Poly.

Polytechnic Institute of Brooklyn has been awarded a grant of $375,000 by the National Science Foundation to support the construction of graduate level research laboratories. They will be established in Polytechnic's Long Island Graduate Center, now under construction in Farmingdale, N. Y. The center is designed to provide graduate education and research in aeronautics, astronautics, electrical engineering, electrophysics, mechanical engineering, mathematics and physics.

The center will consist of two buildings. The main building will contain nine classrooms, a science library for 20,000 volumes, administrative offices, dining area, lounge and meeting room, as well as twelve laboratories for research. The second building will house three laboratories for basic research in aerodynamics and high power electronics. The main building is scheduled for completion in September 1961; the second in March.

Steel Founders' Contest

The Steel Founders' Society of America is sponsoring an expanded Product Development Contest in conjunction with the steel casting industry's centennial observance in 1961. Prizes totaling $10,000 will be awarded in the contest, sponsored by the society in its search for the development of steel casting applications through engineering analysis and design.

Almost any new development or improvement in the use of steel castings will be eligible in the contest which runs to June, 1961. Twenty two awards in each of the five fields will be made in the contest. There will be two classes of entrants: industry and student. Industry personnel, except steel foundry employees and S.F.S.A. staff members, are eligible to compete in the industry class and fulltime students (including graduate students) are eligible to compete in the student class.

Entries for both classes must be made in one of five divisions: 1) end cost, 2) mechanical design, 3) metallurgical design, and 5) unusual or unique.

Complete rules and format guides for the contest are available from the Steel Founders' Society of America, 606 Terminal Tower, Cleveland 13, Ohio.

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SALES POSITION

The Allvac Metals Company, producer of induction vacuum melted high temperature alloys, alnico and ceramic permanent magnets has sales positions open.

QUALIFICATIONS: B.S. Degree in engineering. To 30 years of age. Free to travel. Willing to contribute to and share in the growth of youngest specialty metals company in the U.S.

CONTACT: William O. Nisbet, V. President, Box 611 Monroe, N. C.

INTERNATIONAL NICKEL
Has An Immediate Opening In
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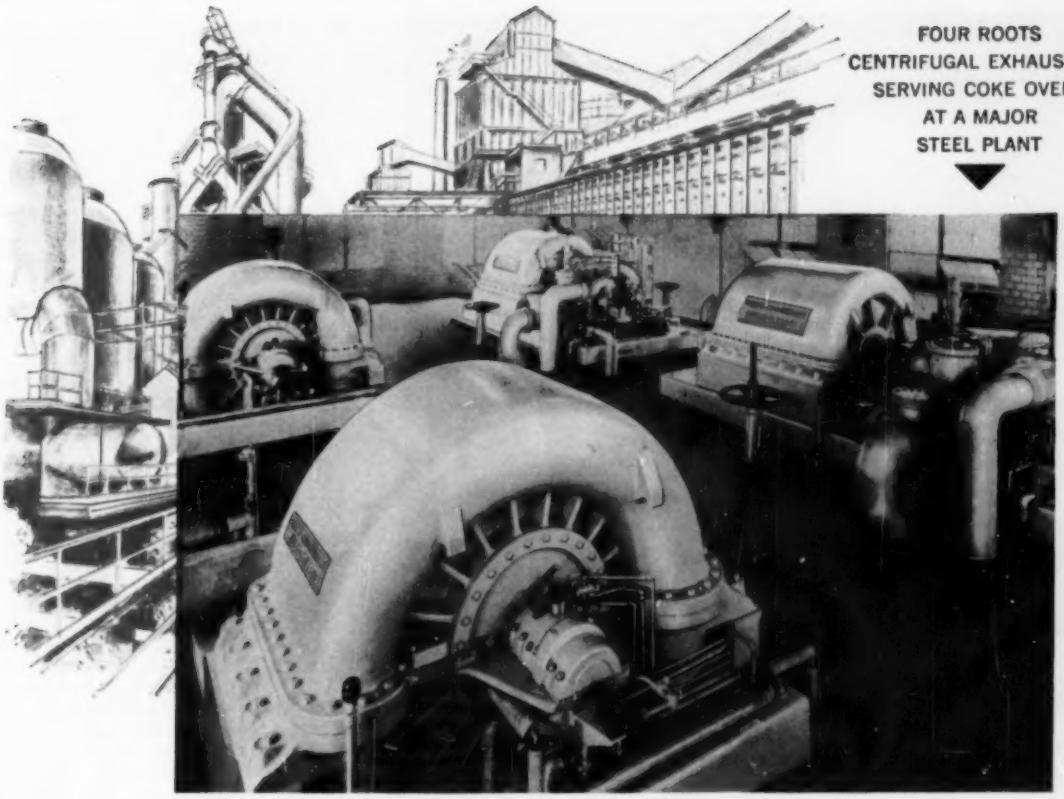

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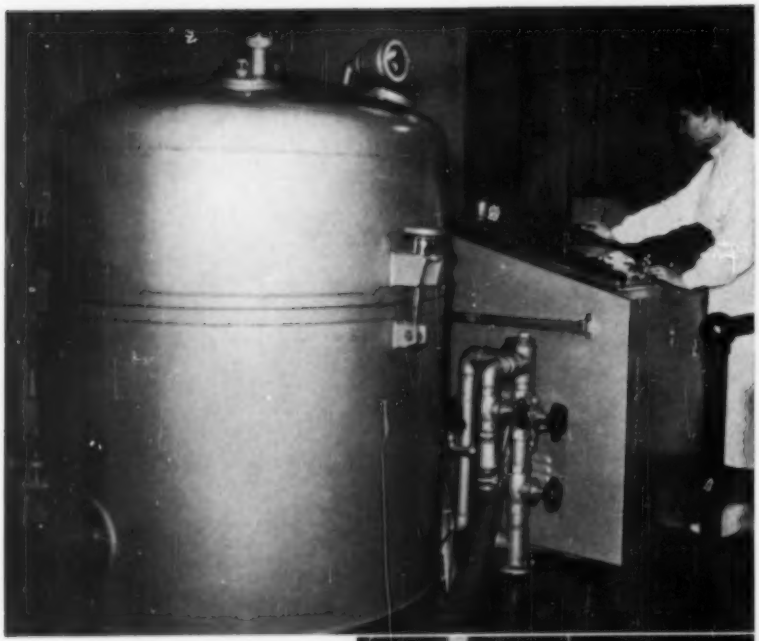

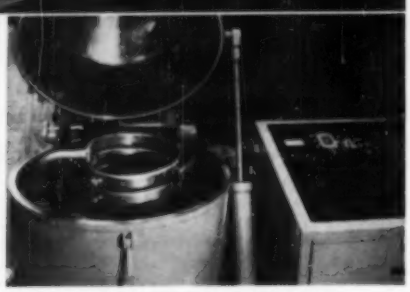

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New Books

(Continued from page 6)

treated. An appendix contains phase diagrams and detailed index. •

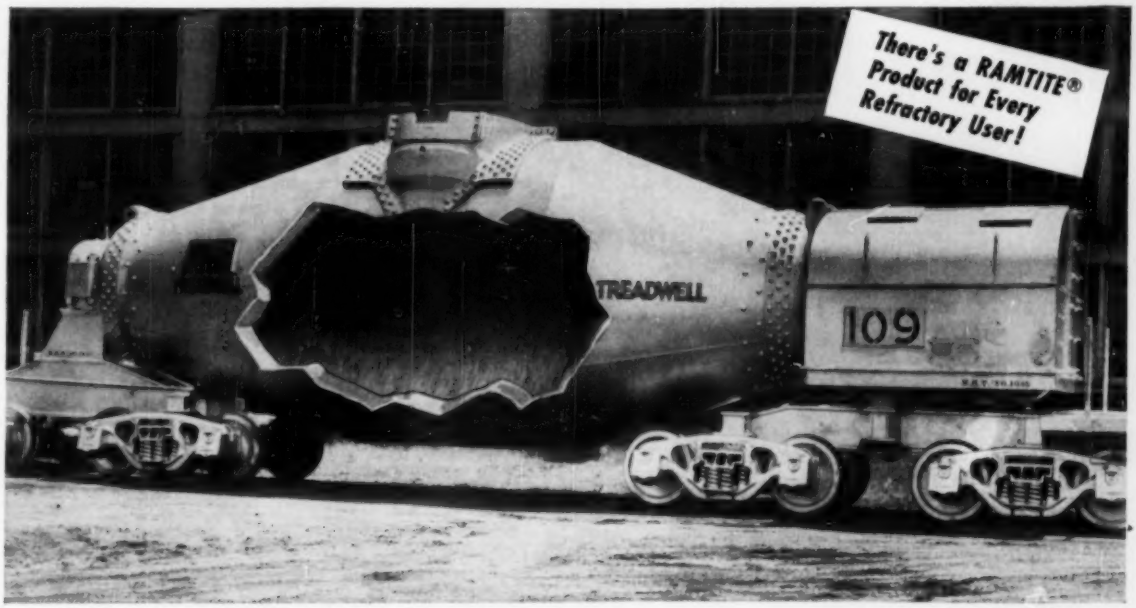
Embrittlement by Liquid Metals, by W. Rostoker and others, Reinhold Publishing Corp., 162 pp., $7.95, 1960 —This book describes embrittlement by liquid metals, and advances a thesis on its fundamental significance, treating the subject as a scientific phenomenon, but pointing out its destructive nature from an engineering viewpoint. The first chapter is a review of the literature on the subject. Succeeding chapters discuss liquid metal embrittlement from the standpoint of its occurrence, influencing factors, delayed failure, and mechanism, as well as crack propagation and the theory of fracture of metal. •

Progress In Powder Metallurgy—1960, Metal Powder Industries Federation, New York, 215 pp., $10, 1960 —This volume contains all technical papers presented at the Federation's Sixteenth Annual Technical Meeting last April. Several new developments in powder metallurgy are covered, including papers devoted to the application of non-destructive testing techniques on powder metallurgy products. The powder metallurgy of nickel, nickel alloys, and stainless steel is covered. Machinability characteristics of powder metallurgy products and low temperature impact strengths of sintered steels are given. Besides properties, the book describes how to get the most benefit from powder metallurgy parts from an applications engineering point of view. The book also contains a report on dispersion strengthening of metals by powder metallurgy methods. •

Progress in Astronautics and Rocketry, Volume 1, **Solid Propellant Rocket Research,** edited by Martin Summerfield, published by Academic Press, Inc., New York. A selection of technical papers based mainly on a Symposium of the American Rocket Society held at Princeton University, January 28-29, 1960. $6.50. •

Non-Crystalline Solids, edited by V. D. Frachette, published by John Wiley & Sons, Inc., New York, covers the proceedings of the Conference on Non-Crystalline Solids which was held in Alfred, N. Y., on September 3-5, 1958. $15.00. •

The Surface Chemistry of Metals and Semiconductors, edited by Harry C. Gatos, with J. W. Faust, Jr., and W. J. LaFleur, published by John Wiley & Sons, Inc., New York. This volume contains the papers presented at the Joint Symposium of the Corrosion and Electronics divs. of the Electrochemical Society which was held in Columbus, Ohio, on October 19-21, 1959. $12.50. •



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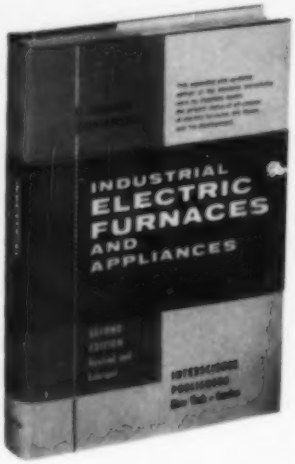

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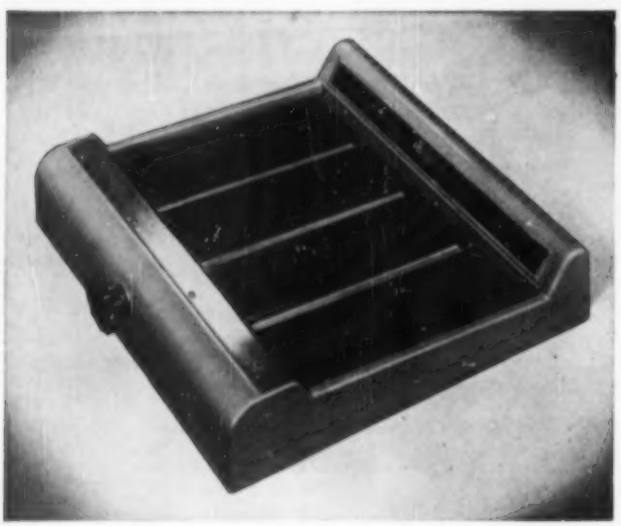

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New fuel cell

A new type of fuel cell, which will be used to develop a chemical power plant for Naval use, was disclosed by The M. W. Kellogg Co., a subsidiary of Pullman, Inc.

The cell operates on a mercury amalgum of sodium, oxygen, and water. This cell rather than a hydrogen and oxygen cell was selected for two basic reasons. First, each cell will develop twice the voltage of the hydrogen cell, thus fewer cells will be required for a sizeable power plant. Secondly, fuel storage requirements will be considerably smaller, since sodium is much denser. Furthermore, sodium fuel is more safely handled than hydrogen.

Like all fuel cells, the sodium-amalgum and oxygen cell is a continuous-feed primary electric battery in which the energy from a chemical reaction is converted directly to electrical energy rather than appearing as heat. The prototype will develop about 100 hp or 75 kw. Together with fuel storage space, it will be about 15 times lighter than the standard electric batteries now in use, and will weigh between 2 and 3 lb per kw-hr of capacity. The efficiency of energy conversion is high—approx 60 pct.

On-off magnetic properties

Scientists of the Du Pont Co. announced the discovery of a new metallic compound which exhibits unique on-off magnetic characteristics at the sixth annual Conference on Magnetism held in New York.

The new compound—chromium manganese antomide—becomes magnetic as the temperature rises above a predetermined point, and when the temperature drops below the point it becomes nonmagnetic. Chemical composition of the alloy determines at which point this occurs, and it was discovered that, by slight changes in composition, the point could be varied over a wide range of temperatures.

The abrupt magnetic change occurs because the compound's inter-atomic distances determine how the magnetic forces align. Temperature variations change the distance between atoms, hence change the magnetic properties.

Esso's oil injection

Possibility of lower-cost pig-iron production by injection of heavy fuel-oil into blast furnaces was announced by Esso Research & Engineering Co., an affiliate of Standard Oil Co., N. J. The key to Esso's development apparently lies in the design of the injection and control system. In arriving at a suitable system, Esso, designed, built, and operated a pilot-plant furnace. The system was later installed on an operating, commercial furnace with complete success. Esso research is continuing work on experimental—and full-scale furnaces to obtain more data.

Injection in UK and USSR

British Iron and Steel Research Association is (BISRA) is now planning full-scale trials of blast-furnace injection in cooperation with the National Coal Board and the oil companies. Efforts are being made to take advantage of the use of oil, powdered coal, or natural gas for increasing blast furnace production rates while lowering operating costs. Increasing furnace efficiency has already reduced coke consumption 33 to 34 pct per ton of iron.

In the Soviet Union, a chairman of the State Committee for Science and Technology stated that Soviet blast furnaces produce 30 to 35 pct more iron than do US furnaces of comparable capacity. The Soviet Government is also ready to make known, under license, the method of using natural gas in blast furnaces, "since no other country is in possession of the secret so far". [? Ed]

Natural gas is currently used at the Dzerzhinskiy iron and steel works, resulting in the production of the Ukraine's lowest-cost pig iron.

Urals Ferrous Metals Institute has evolved a method of firing blast furnaces with refinery waste products such as mazout—a low-grade fuel oil—since natural gas is not yet available in that area. This method resulted in considerable coke savings, and increased pig iron output by 14 pct when used at the Chuseyer metallurgical works.

US Steel injects gas

American Metal Market reports that US Steel has been injecting some 3 million cu ft of natural gas per day into one of its blast furnaces at the Fairless works. The furnace has reduced coke consumption from 1400 lb per THM to about 1160 lb while increasing output by 10 pct. The Fairless experiment determined that for each increase of 100°F in blast temperature approx 30 lb of coke per THM could be saved. It is also reported that Colorado Fuel & Iron and McClouth Steel are conducting experiments on gas injection.

Experimental blast-furnace

Officials of U. S. Steel Corp, announced that work is progressing on the building of a small experimental blast furnace at the Corp. Universal Atlas Cement div. plant at Universal, Pa.

The experimental unit will further research studies of the chemical changes experienced by raw materials during reduction. Furnace design enables it to operate at much higher pressures, temperatures, and larger blast air volumes than a conventional furnace. It is designed for top pressures of 50 psi, and the 5000 cfm of blast will be heated to a maximum of 2500°F. Fully-automatic probes will enable the researchers to record gas composition and temperatures, analyze top gases, and take pressure readings over the entire length of the stack during operation. Hearth diameter of the experimental furnace will be 4 ft, and its working height from tuyeres to stockline will be 19½ ft.

REPORTER

New French Al Process

Recent reports that Pechiney and Ugine have developed a new aluminum process were confirmed recently by Pechiney in Paris.

The new process involves the carbothermic reduction of aluminum oxides yielding a metal of normal purity, but with a much lower titanium content than obtained by normal practice. The process consists of two stages: 1) production of pure corundum in an electric furnace by carbo-thermic reduction of bauxite; and 2) further reduction of the corundum in an electric furnace in the presence of carbon. The separation of aluminum from the metal-carbide mixture is thus obtained, and the aluminum carbide remaining can be recycled for subsequent reduction.

Preliminary tests were performed in a 120-kva furnace, and subsequent tests have been run in 700- and 3000-kva furnaces. A plant currently making pilot runs at Nogueres is expected to be in full operation early in 1961 at an annual capacity of 3000 to 5000 tons.

The first patents relating to this process were registered in 1951, and at the present time, patents applied for in principal countries have either been granted or are in the process of being examined.

Irkutsk Aluminum Plant

The Metal Bulletin (November 8, 1960) reports that a giant aluminum plant is under construction in the Irkutsk region of Siberia in the USSR. Construction of the plant began three years ago. It contains an electrolysis shop which is almost a third of a mile long. The electrolytic cells are scheduled to begin operating next year. The plant will be the largest of its kind in the USSR. When operating at full capacity, the plant will reportedly produce as much aluminum as is produced by all the aluminum plants of France and Italy combined (this would place the annual production at more than 250,000 metric tons). The plant also contains a thermal ore furnace (sic), which has begun operation.

New columbium sources

Commercial production of pyrochlore columbium concentrate containing a minimum of 50 pct Cb_2O_5 is scheduled to begin during the summer of 1961 from a major new source of the metal. The concentrates will be processed at St. Lawrence Columbium & Metals Corp.'s mines at Oka, located 20 mi from Montreal. Initial mill capacity is expected to be 500 tpd. Meanwhile, the company is supplying samples to various producers from its pilot-plant mill which has been operating since December 1959.

At the same time, Molybdenum Corp. and Wah Chang are now building a large plant in Brazil to concentrate ores from what is believed to be the world's largest columbium deposit.

Electrolytic machining

An electrolytic machining process that can shape parts from the toughest alloys in as little as one-tenth of the time required by conventional machine tools has been developed by Battelle Memorial Institute in research supported by the Steel Improvement & Forge Co. of Cleveland. To produce a part, a rough forging or piece of metal stock (anode) is placed between shaped electrodes (cathode), and electrolyte is pumped under pressure into the space between the electrodes and the workpiece. As the electric current dissolves the workpiece to the desired shape, the electrodes are moved in simultaneously. The electrolytic unit has machined rough forged jet turbine blades to 0.003 in. tolerances in 5 to 10 min—an operation which took from 1 to 2 hr to perform by grinding. Other types of units can be used to sink die cavities and drill round and irregularly-shaped holes in alloys that cannot be penetrated by conventional drills.

Current requirements for the process range up to 1500 amp per sq. in. of workpiece at 3 to 12 v, to achieve a penetration rate of 0.05 to 0.1 in. per min. However, for most operations 100 to 500 amp is sufficient.

Another Kaldo

In England, Tube Investments has announced a major expansion program for its subsidiary, Park Gate Iron & Steel Co. A new $165-million works of some half million ton ingot capacity is to be built on a site adjacent to the existing plant. It will comprise an ore preparation plant, a blast furnace, a steel-making shop with Kaldo and electric furnaces, and rolling mills. There are to be two Kaldo furnaces, each of about 80 ton capacity. Park Gate will be the fifth British firm to adopt one of the oxygen steelmaking processes, and the second in Britain to take up the Kaldo method.

Inco ore plant expands

The International Nickel Co. of Canada is undertaking a $50-million expansion program of its iron-ore recovery plant at Copper Cliff, Ont., Canada. The expansion, scheduled for completion in three years, will triple present facilities.

Using a process developed by the company's research staff, the expanded plant will treat 1.2 million tons per year of nickeliferrous pyrrhotite. Nickel is removed by atmospheric leaching, and the process also involves solid-state pyrometallurgical operating in high-capacity units. By diverting this large quantity of pyrrhotite to the expanded plant, a 40-pct decrease in the tonnage of materials otherwise handled by the nickel section of the smelter is expected. This change will result in important economies.

In the iron-ore recovery plant, capacity of iron pellets, containing 68 pct Fe, will be raised to 900,000 tons per year, and more nickel will be recovered from comparable amounts of pyrrhotite than is now possible.

For a description of the process, see the August 1958 issue of JOURNAL OF METALS, p. 527.

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editorial

Where are the Blast Furnaces Going?

For the past months, JOURNAL OF METALS has been publishing considerable material on the development of various direct iron-reduction processes. Other than the Holowaty-Austin article *Quo Vadis Blast Furnace?* in the April 1960 issue, little has appeared in these pages on the giant of iron production, the blast furnace. But, as the sun continues to rise and set, so today's monster blast furnace continues to pour forth an endless stream of hot metal for the industrialized world.

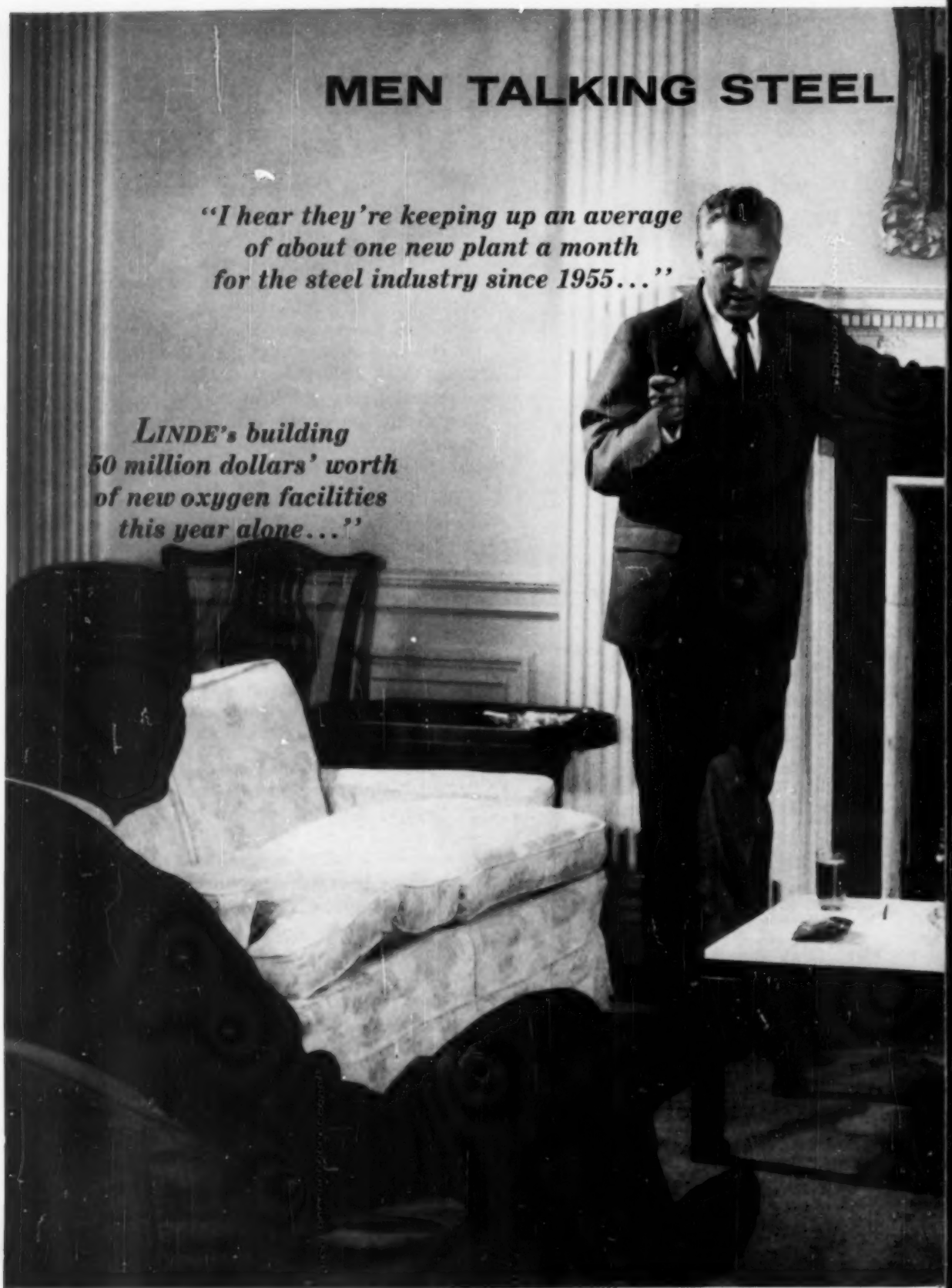
For decades, few changes could be noted in the mammoth shaft furnace—it being some time since charcoal had given way to anthracite and then coke and the introduction of the hot blast. But, in the past few years, something has begun to change as increasing demands were put on the furnace in response to higher operating and capital costs, coke shortages, and greater demands for iron. Some of these changes have been in the direction of automation of operations, while design changes have included provision for higher top pressures, controlled humidity, etc.

In this issue we emphasize another approach to improved blast-furnace performance—injections through the tuyeres. This has been an area of intense experimental work in many iron-producing countries. These injections—including oxygen, natural gas, coke-oven gas, various other industrial gases, steam, nitrogen, fuel oil, pulverized coal, and other materials—have been accompanied by experimental use of higher blast temperatures and top pressures. Tests have shown the possibility of considerably increased output accompanied by reduced coke requirements. Results have been so successful that industrial blast-furnace operation with injections of gases and liquids is now being undertaken. General adoption by leading iron producers seems assured.

This line of development, together with a more intensive campaign for burden preparation, bodes well for the future of the blast furnace.

Quo Vadis Blast Furnace?—well, it looks as if it will be around for quite some time.

FWS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High-strength steel
19" diameter
143 pounds

PHYSICAL PROPERTIES

	ALLOY*	TEST TEMPERATURE °F.	HEAT TREAT	GUARANTEED MINIMUM ULTIMATE TENSILE STRENGTH PSI	GUARANTEED MINIMUM 0.2% YIELD STRENGTH PSI	GUARANTEED MINIMUM ELONGATION %	TYPICAL VALUES ULTIMATE TENSILE STRENGTH PSI	TYPICAL VALUES 0.2% YIELD STRENGTH PSI	TYPICAL VALUES ELONGATION %
TENSILE AND YIELD STRENGTHS	Waspaloy	RT	A	175,000	120,000	15.0	194,000	136,000	24.0
		1000	A	162,000	112,000	13.0	175,000	124,000	23.0
	René 41	RT	B	180,000	132,000	12.0	200,000	150,000	15.0
		1400	B	135,000	115,000	13.0	145,000	125,000	18.0
	Astroloy	RT	C	190,000	138,000	10.0	205,000	152,000	12.0
		1400	C	150,000	122,000	12.0	160,000	132,000	15.0
	U-500	RT	D	175,000	120,000	10.0	195,000	135,000	12.0
		1400	D	125,000	105,000	12.0	140,000	120,000	15.0
	M-252	RT	B	170,000	115,000	15.0	185,000	125,000	20.0
		1000	B	160,000	105,000	15.0	165,000	115,000	22.0

	ALLOY*	TEMPERATURE STRESS	GUARANTEED MINIMUM	TYPICAL VALUES
STRESS RUPTURE	Waspaloy	1350°F.—70,000 psi	70 hours—6% Elongation	130 hours—16% Elongation
	René 41	1350°F.—85,000 psi	30 hours—8% Elongation	60 hours—15% Elongation
	Astroloy	1800°F.—20,000 psi	20 hours—7% Elongation	35 hours—12% Elongation
	Udimet 500	1650°F.—25,000 psi	30 hours—6% Elongation	55 hours—12% Elongation
	M-252	1500°F.—40,000 psi	30 hours—8% Elongation	55 hours—16% Elongation

NOTE: The guaranteed minimums are based upon radial and tangential test locations.

COMPOSITION AND HEAT TREATMENT

	ALLOY*	ELEMENTS C	Cr	Mo	Co	Ti	Al	B	Ni
CHEMISTRY	Waspaloy	.08	19.0	4.3	13.5	3.0	1.4	.004	Balance
	René 41	.08	19.0	10.0	11.0	3.1	1.6	.004	Balance
	Astroloy	.05	15.0	5.0	15.0	3.5	4.3	.030	Balance
	Udimet 500	.07	18.5	5.0	18.3	3.0	3.0	.003	Balance
	M-252	.15	20.0	10.0	10.0	3.0	1.3	.002	Balance

HEAT TREATMENT	A.	1850°F.—4 hours—OQ; 1550°F.—2 hours—AC; 1400°F.—16 hours—AC
	B.	1950°F.—4 hours—AC; 1400°F.—16 hours—AC
	C.	2100°F.—½ hour—AC; 1400°F.—16 hours—AC
	D.	1975°F.—4 hours—AC; 1500°F.—24 hours—AC; 1400°F.—16 hours—AC

NOTE: Refer to appropriate heat treat code in Tensile & Yield Strength table.

*Waspaloy: Pratt & Whitney Aircraft Corp.; René 41, Astroloy and M-252: General Electric Company; Udimet 500: Metals Division, Kelsey-Hayes Company.

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WASPALOY

●

RENÉ 41

●

ASTROLOY

●

UDIMET 500

●

M-252

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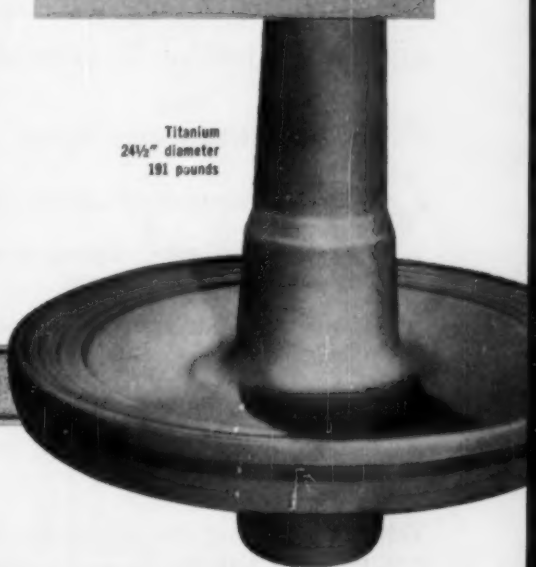
Titanium
15½" length

EST. 1903

FORGED



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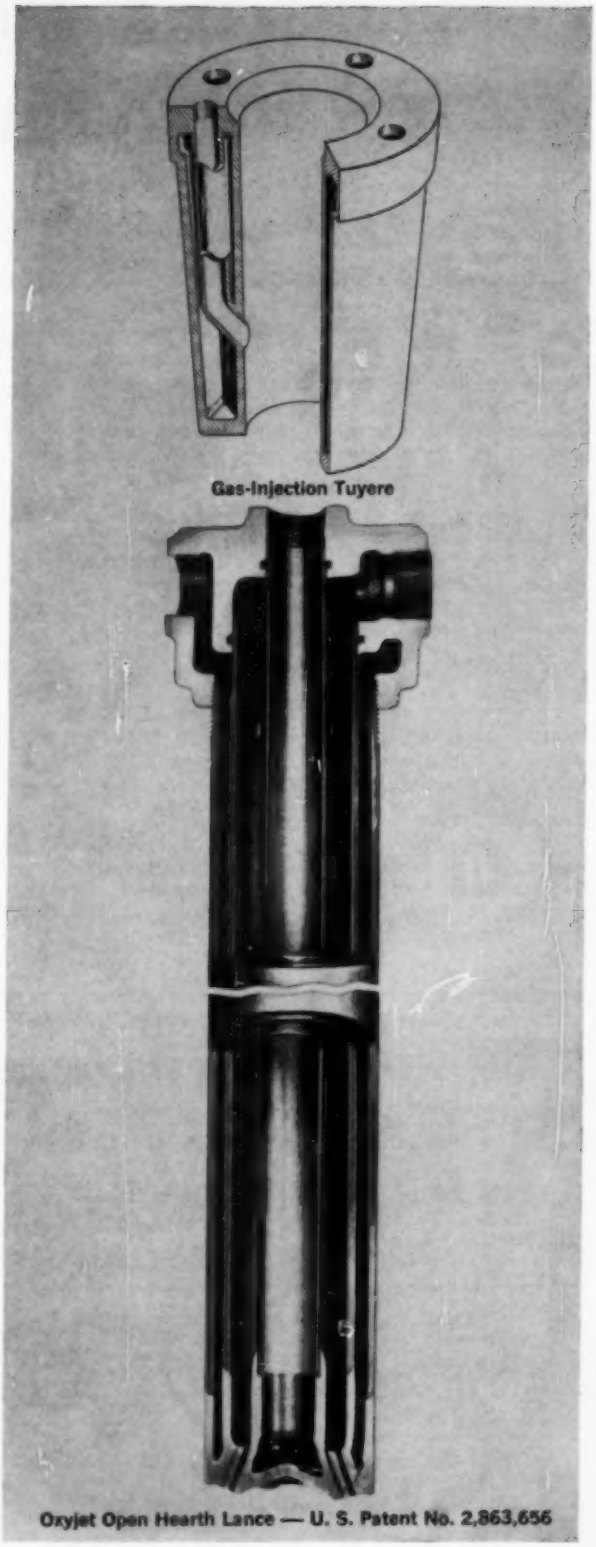

Gas-Injection Tuyere

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BLAST FURNACE ENRICHMENT INVESTIGATIONS

Results of investigations carried out in the US Bureau of Mines' experimental blast furnace at Bruceton, Pa. are reported. The effects of moisture, natural gas, and oxygen injection are described.

by E. J. Ostrowski, G. Kesler, and N. B. Melcher

Successful experimental work has been done by the US Bureau of Mines in cooperation with the National Steel Corp., using gaseous additions to the air blast and also to the bosh of the Bureau's experimental blast furnace at Bruceton, Pa. Steam, natural gas, and natural gas plus oxygen injection were studied. Each of these additions required additional blast temperature, and the use of this higher blast temperature resulted in a decreased coke rate and an increased metal production rate. Additional coke savings were realized when the carbon from the natural gas replaced carbon from the coke.

The principal dimensions of the experimental blast furnace and one of the two blast preheat stoves are shown in Fig. 1. This furnace is equipped with a 4-ft diam hearth, which is suspended and which can readily be detached. Blast air enters the furnace through three water-cooled copper tuyeres, spaced at 120°. The bosh is carbon-lined, and the remainder of the furnace, as well as the hearth, is lined with super-duty fire brick. Two bells and a revolving hopper at the top insure good stock distribution, and two off-takes discharge the top gases. The stoves preheating the blast are lined with high-alumina refractory in the hot zone. Alumina spheres approx 1 in. in diam form the bed which acts as a heat exchanger. The stoves are fired downward using natural gas and air. With this construction, it is possible to operate at a blast temperature above 2600°F. The bustle pipe and the blowpipes are refractory lined to withstand a temperature of 3000°F. Fig. 2 shows the location of the auxiliary and bosh tuyeres used for injecting the natural gas. These auxiliary tuyeres are of simple design and are constructed of standard copper pipe and fittings.

The method used for injecting natural gas through the main tuyere is shown in Fig. 3, and the metering and control system used is illustrated in Fig. 4. Moisture additions to the blast were accomplished by injecting steam into the blast ahead of the stoves, and oxygen enrichment of the blast was accomplished in much the same manner. The control and metering system used for steam injection is shown in Fig. 5.

The experimental program at Bruceton was conducted on a burden similar to that used on the D furnace of the Great Lakes Steel Corp., in Detroit, Mich. To maintain the specified percentages of iron-bearing materials, 70 pct sinter, 17 pct Hanna coarse ore, and 13 pct Weir coarse ore were used in a 10-round ore-flux-coke charging cycle. All materials were charged on a dry weight basis. The sinter and the Hanna coarse ore were screened at + 10 mesh prior to being loaded into the skip. Table I shows the typical screen analysis of the materials used, and Table II shows the chemical composition of the burden materials. The coke used contained the

Table I. Typical Screen Analysis of Sinter and Ores, pct

Screen size	Sinter	Hanna ore	Weir ore
+ 1 to 1½ in.	2.2	35.8	35.2
+ ½ to 1 in.	12.2	41.4	29.4
+ 3 mesh to ½ in.	43.2	6.4	10.8
+ 10 mesh to 3 mesh	37.6	3.0	9.4
+ 65 mesh to 10 mesh	2.4	1.0	5.4
− 65 mesh	3.4	12.4	9.8
Total	100.0	100.0	100.0

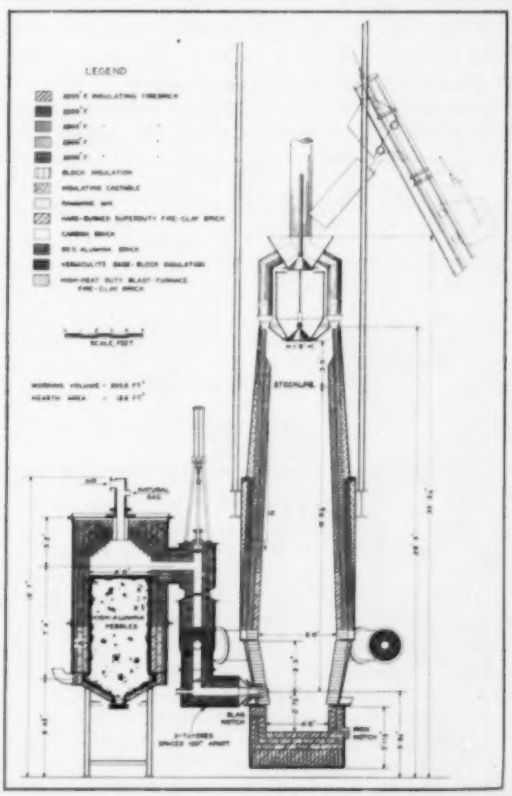


Fig. 1—Cross-section of experimental blast furnace and pebble stove.

E.J. OSTROWSKI and N. B. MELCHER are with the US Bureau of Mines, and G. KESLER is with National Steel Corp., Weirton, W. Va. This paper was presented at the 1960 AIME Blast Furnace, Coke Oven, and Raw Materials Conference.

Table II. Chemical Composition (Dry Basis) of Burden, pct

	Fe	MgO	P	SiO_2	Al_2O_3	CaO	TiO_2	C	FeO	Fe_2O_3
Hanna ore	54.47	0.25	0.10	12.31	2.08	1.63	0.06	—	1.42	76.30
Weir ore	55.14	1.13	0.45	7.87	3.94	1.74	0.08	—	3.80	74.62
Sinter	54.20	1.32	0.14	11.32	2.68	7.99	0.07	—	10.96	65.28
Coke ash	9.72	1.71	—	44.08	27.13	6.0	1.51	—	—	—
Calcite limestone	0.61	0.49	—	1.82	1.33	54.40	—	—	—	—
Dolomite	1.65	21.06	—	0.46	1.20	26.69	—	—	—	—

following: 1.5 pct volatile matter, 89.6 pct fixed carbon, 8.6 pct ash, and 0.65 pct S. Natural gas used in the injection contained 90 pct CH_4, 6.1 pct C_2H_6, 1.9 pct C_3H_8, 0.8 pct C_4H_{10}, 0.9 pct N_2, and 0.1 pct CO_2.

The wind rate was held at 800 std cfm except in periods when it was reduced to maintain a constant bosh gas volume. The base level of moisture in the air blast was 7 grains per cu ft.

Greater heat losses per unit of production in the experimental furnace as compared to a commercial furnace necessitated higher blast temperatures on the order of 350 to 450°F.

The investigation was begun by operating the experimental furnace for a period of 122 hr on a Great Lakes burden with an ore-to-coke ratio of 2.30 to 1. The purpose of this test period was to stabilize operating conditions and to ascertain the degree to which the operation and results obtained on the experimental furnace would simulate the required standard imposed by the practice of the industrial furnace. These standards were:

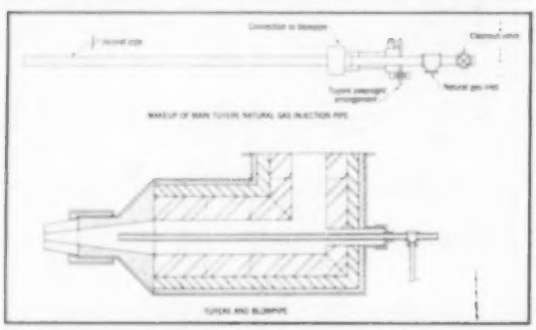
1) Silicon in the metal, 0.90 pct;
2) Sulfur in the metal under 0.40 pct;
3) Slag basicity to range from 1.02 to 1.10;
4) Slag rate per ton of hot metal approx 1000 lb; and
5) MgO content of slag to range from 10 to 11 pct.

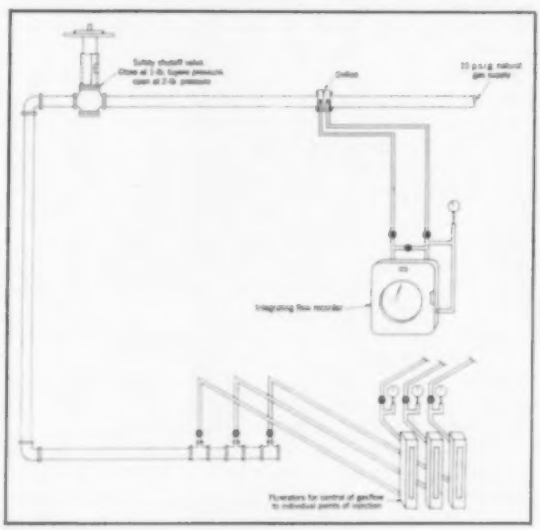
The results of this test (No. 1) are shown in Table III. This Table also shows the results of all other test periods, and throughout this paper reference will be made to these test periods only. The values given in the Table are averaged over the number of operating hours. Transition periods between tests are not included, and data gathered during such a period are not a part of the reported results.

In Test 2, the minimum operable coke rate for the burden being used was established. This was accomplished by decreasing the coke rate and increasing the blast temperature to a point where specification-grade iron could not be produced, and additional blast temperature caused the burden movement to become erratic and the furnace tended to hang. The minimum coke rate attained was 1417 lb per ton of hot metal (THM) at a blast temperature of 1800°F. With this period, a base or reference point was established to which other tests could be related.

Increased moisture

Tests 3, 4 and 5 constituted the studies of increased moisture in the blast. Test 3 was conducted at a fixed blast temperature of 2100°F. The wind blowing rate was adjusted to give a constant bosh gas volume. This adjustment varied, depending on the amount of moisture required to produce quality metal, and a





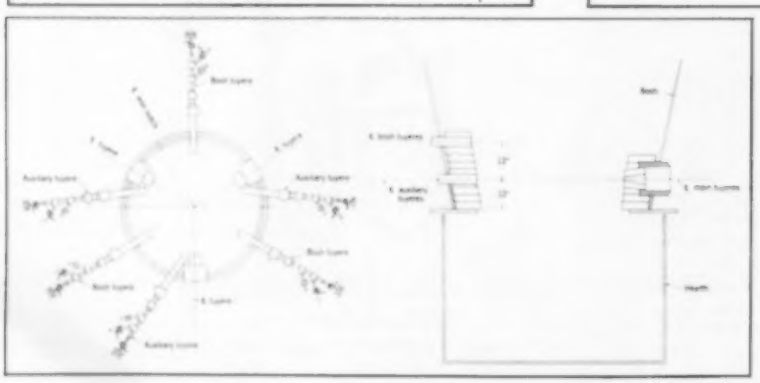


Fig. 2—Left, Arrangement and location of main, auxiliary, and bosh tuyeres. Fig. 3—Upper left, Section showing refractory lined blow pipe, tuyere, tuyere cooler, and Inconel pipe for injection. Fig. 4—Above, Arrangement of piping, metering, and control for natural gas injection.

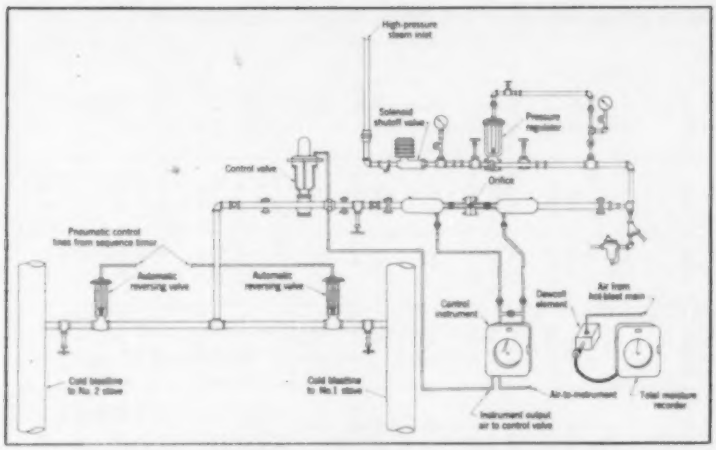


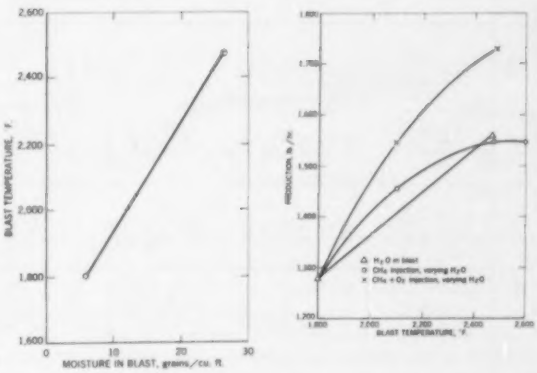
Fig. 5—Left, Arrangement of piping, metering, and control for moisture injection. Fig. 6—Below left, Hot-blast temperature vs H_2O in blast. Fig. 8—Below right, Production rate vs hot-blast temperature.

coke reduction per charge was made before the moisture was added. Under these conditions, an avg of 13 grains of moisture per cu ft of air was used. The aim of Test 4 was comparable, but operations were carried out at a blast temperature of 2475°F. A further coke reduction was made. The average moisture in the blast was 33 grain per cu ft of air. In Test 5 the wind rate was increased and held constant at 800 std cfm. No changes in the ore-to-coke ratio or blast temperature were made. Optimum results were obtained with 26 grains of moisture per cu ft of air. Comparison of tests 3, 4, and 5 to the base period showed a decrease in the coke consumption per THM of 38, 103, and 88 lb and an increase in metal production per hr of 78, 134, and 280 lb, respectively. Furnace operation was smooth and the aims of the program were generally achieved. Only a few casts were higher in sulfur than specified.

Natural gas injections

Tests 6, 7, and 8 were run to study methods of introducing natural gas into the experimental blast furnace. In Test 6, the gas was injected through the auxiliary tuyeres adjacent to the main tuyeres. For Test 7 the gas was injected through the main tuyeres, and in Test 8 through the bosh tuyeres. Before natural gas was injected, a decrease in coke per unit charge was made. This change in coke weight was made 7 hr prior to introducing the gas, this being the length of time required for the change in burden to become effective at the tuyeres. The blast temperature was held constant at 2475°F. As the changes in burden reached the tuyeres, increasing amounts of natural gas were injected, and the moisture of the blast was decreased until conditions became stabilized at 7 grains. Thereafter, operations were controlled by adjusting the rate of natural gas input. Injection through the auxiliary tuyeres in Test 6 gave good furnace performance; however, some of the natural gas escaped at the juncture of the hearth and bosh, and the amount of CH_4 in the top gas increased. The average gas injection per tap of hot metal for this test was 3476 std cu ft.

A further reduction in the coke charge was made for Test 7. Other conditions remained the same as in Test 6, except that injection was through the main tuyeres. Utilization of the gas, as indicated by the top gas analysis, was practically complete, and there was no evidence of leakage at the hearth-bosh juncture. A total of 3243 std cu ft of natural gas was injected per THM.


BLAST TEMPERATURE, °F
MOISTURE IN BLAST, grains/cu. ft.
PRODUCTION, lb./hr.
BLAST TEMPERATURE, °F
Δ H2O in blast
O CH4 injection, varying H2O
× CH4 + O2 injection, varying H2O

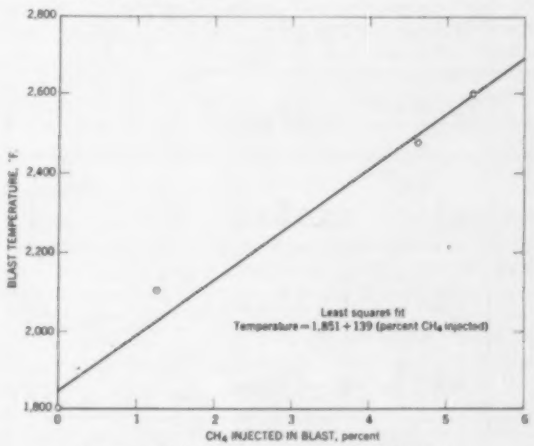
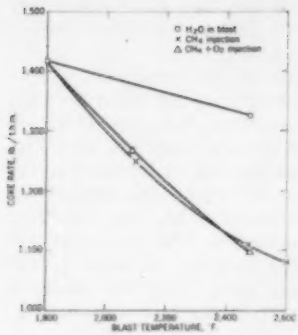


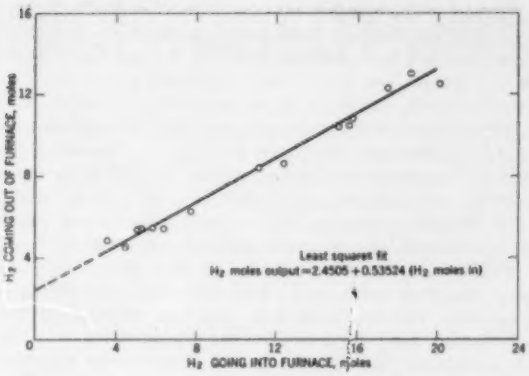
Fig. 7—Hot-blast temperature vs CH_4 injected.

Before natural gas was injected through the bosh tuyeres, Test 8, the coke weight per charge was increased from the 154 lb used in Test 7 to 168 lb. The blast temperature remained at 2475°F, and no change in moisture was made. The injection of natural gas was changed abruptly from the main tuyeres to the bosh tuyeres. The furnace almost immediately went into a hang; consequently, injection into the bosh tuyeres was discontinued and was begun through the auxiliary tuyeres. The following day, after the furnace had operated normally for approx 20 hr, injection into the bosh tuyeres was begun again. This time the transition was done gradually, i.e., the gas flow through the auxiliary tuyeres was decreased while that through the bosh tuyeres was

Table III. Average Operating Data for Natural Gas, Steam, and Natural Gas Plus Oxygen Injection

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Hours of operation	120	64	72	96	96	48	81	100	122	124	55	47	47	51	48
Total rounds	517	283	336	468	523	278	429	611	721	750	298	253	236	253	256
Rounds per hour, average	4.31	4.4	4.67	4.88	5.45	5.79	5.30	6.11	5.90	6.05	5.42	5.38	5.02	4.96	5.33
Burden ratio: $\frac{Ore}{Coke}$	2.30	2.44	2.50	2.63	2.63	3.13	3.25	2.98	3.23	3.18	3.16	3.26	2.75	2.75	2.73
Material per round, percent:															
National sinter	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70
Hanna ore	17	17	17	17	17	17	17	17	17	17	17	17	17	17	17
Weir ore	13	13	13	13	13	13	13	13	13	13	13	13	13	13	13
Coke, pounds per round	217	205	200	190	190	160	154	168	155	157	158	153	182	182	183
Dolomite, pounds per round	52	50	51	50	50	50	50	52	52	52	52	52	52	52	52
Limestone, pounds per round	21	16	19	15	15	15	15	15	15	15	15	15	15	15	15
Dolomite injection	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Limestone injection	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Grains H_2O per cu. ft. air	7.0	6.1	13.4	33.0	26.6	7.0	7.0	7.0	7.0	6.2	5.5	3.2	4.6	3.3	3.4
Moisture, percent	2.20	2.0	4.35	9.31	7.60	2.20	2.20	2.20	2.20	2.00	1.80	1.00	1.50	1.0	1.0
Std. c.f.m. of CH_4	-	-	-	-	-	47.6	40.7	23.1	34.2	37.3	37.2	43.1	10.1	11.2	11.2
CH_4, percent	-	-	-	-	-	5.95	5.1	2.92	4.31	4.56	4.64	5.35	1.27	1.47	1.40
Std. c.f. of CH_4/THM	-	-	-	-	-	3470	3243	1594	2498	2587	2892	3356	826	940	886
Std. c.f.m. of O_2	-	-	-	-	-	-	-	-	-	20	-	-	-	-	5.6
O_2, percent	-	-	-	-	-	-	-	-	-	2.44	-	-	-	-	.68
Std. c.f. of O_2/THM	-	-	-	-	-	-	-	-	-	1386	-	-	-	-	435
Wind blowing rate (std. c.f.m.)	792	787	762	705	777	774	781	783	781	785	785	782	786	765	783
Air/THM (1000 std. c.f.)	76.58	72.83	66.48	59.05	58.04	54.81	60.39	53.64	56.25	52.70	59.38	59.35	64.24	63.16	59.81
Top gas temperature, °F.	504	485	419	350	326	310	432	290	403	360	418	419	440	380	405
Hot blast temperature, °F.	1621	1800	2100	2475	2475	2475	2475	2475	2475	2475	2475	2600	2100	2100	2100
Metal production:															
tons per day	14.77	15.35	16.28	16.95	18.70	19.76	18.30	20.84	19.82	20.77	18.51	18.55	17.46	17.18	18.54
Slag rate: pounds/THM	1062	1055	1020	971	978	933	1020	999	980	980	949	957	993	963	988
Coke rate: pounds/THM	1519	1417	1375	1313	1328	1126	1070	1182	1109	1097	1109	1065	1250	1261	1265
Slag basicity: $\frac{CaO + MgO}{SiO_2 + Al_2O_3}$	1.04	1.04	1.11	1.02	1.04	1.02	1.04	1.03	1.04	1.05	1.06	1.05	1.03	1.04	1.01
Metal composition, wt. percent:															
Si	.95	.87	1.16	.90	.87	.85	.75	.76	.66	.83	.79	.82	.82	.83	.68
S	.034	.049	.021	.045	.041	.055	.042	.070	.050	.036	.037	.036	.046	.044	.069
Slag composition, wt. percent:															
Fe	.56	.82	.69	.90	.67	.80	.76	.83	.76	.60	.58	.62	.93	.71	.78
S	.84	.67	.88	.72	.68	.51	.67	.63	.60	.69	.69	.61	.63	.63	.56
SiO_2	36.07	36.91	35.43	37.28	36.88	37.22	37.52	35.06	37.54	37.24	37.15	37.14	37.10	36.66	37.38
Al_2O_3	10.39	10.23	10.32	10.23	10.13	9.93	10.12	10.05	9.73	9.83	10.09	9.79	9.97	10.27	10.41
CaO	37.80	37.51	39.46	37.76	37.13	37.12	37.83	37.19	37.79	38.07	37.98	37.98	38.93	37.50	37.11
MgO	11.02	11.14	11.25	10.60	11.56	11.60	11.26	11.54	11.41	11.61	12.03	11.67	11.09	11.41	11.32
Dry dust, pounds/THM	5.9	6.0	3.8	3.4	3.8	2.4	6.4	4.2	8.0	7.3	7.8	6.8	4.6	5.1	4.7
Top gas analysis, percent by vol.															
CO	25.03	26.20	26.10	25.93	26.40	24.10	23.80	25.32	24.20	26.10	25.20	24.40	25.40	25.80	26.00
CO_2	15.12	13.50	14.90	15.62	15.60	15.30	14.50	15.80	15.70	15.60	14.00	14.00	14.40	14.20	14.50
H_2	1.70	1.90	2.60	4.60	3.90	4.10	5.70	2.20	5.00	5.20	5.50	5.90	2.40	2.40	2.40
O_2	.20	.30	.10	.30	.30	.40	.30	.70	.50	.30	.40	.40	.30	.10	.40
N_2	57.85	58.00	56.00	53.45	53.70	53.60	55.50	54.58	54.40	52.30	54.80	55.30	57.40	57.20	56.60
CH_4	.10	.10	.10	.10	.10	2.50	.10	1.50	.20	.10	.10	.10	.10	.30	.10
Tuyere pressure (p.s.i.g.)	4.5	4.6	4.5	4.5	5.4	5.2	5.5	5.5	5.0	6.3	6.1	6.4	5.0	5.0	5.1





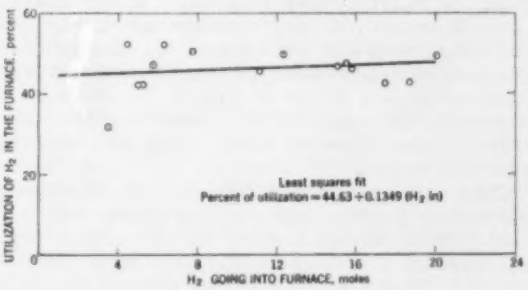

UTILIZATION OF H_2 IN THE FURNACE, percent
Least squares fit
Percent of utilization = 44.63 + 0.1349 (H_2 in)
H_2 GOING INTO FURNACE, moles

Fig. 9—Left, Coke rate vs hot-blast temperature. Fig. 10—Right, Hydrogen in top gas vs hydrogen input. Fig. 11—Below right, Hydrogen utilization in experimental blast furnace.

Table IV. Effect of Blast Composition on Production Rate and Coke Rate

Blast additions	Moisture	Natural gas[a]	Natural gas[a] and oxygen
Wind rate, std cfm	800	800	800
Blast temperature, °F	2,475	2,475	2,475
Moisture grains per cu ft of air	26.6	5.49	6.21
Natural gas, pct	—	4.64	4.56
Oxygen, pct	—	—	2.44
Metal production, tpd	18.70	18.51	20.77
Coke rate, lb per THM	1,328	1,109	1,097

[a] Control with moisture

increased. No difficulties were encountered. The rate of natural gas flow was adjusted to control the operations and metal chemistry. The quantity of gas injected per ton of hot metal was only 1594 std cu ft and of this quantity, only 26.6 pct was utilized. As the results show, the CH_4 in the top gas was high. A comparison of Tests 6, 7, and 8 with the base period test shows an hourly increase in metal production of 368, 246, and 458 lb, respectively. The corresponding coke savings per ton of hot metal were 291, 347, and 235 lb. Control by varying the rate of natural gas injection was not satisfactory as indicated by the low-silicon, high-sulfur content of the casts.

Test 9 was run to reaffirm the results of Test 7; conditions were identical to those of Test 7, except that the average rate of gas injection was unintentionally less, due to wide variations in the gas rate required to control the furnace. This resulted in higher production.

Oxygen enrichment

In Test 10 the blast was enriched with oxygen in sufficient quantity to burn the natural gas to CO and H_2. The injection rate for natural gas was set at 40 std cfm; therefore, 20 std cfm of oxygen was introduced to the blast. The blast temperature was maintained at 2475°F, while the moisture of the blast was varied for control. With the use of moisture for control, very good operating results were obtained. The use of oxygen in the blast resulted in an increased production rate, while the coke rate remained virtually the same. The increase in hourly production was 452 lb, and the coke saving per THM was 420 lb, as compared to the base period.

Temperature variation

Tests 11, 12, and 13 involved the injection of natural gas through the main tuyeres at different blast temperatures. Blast moisture was adjusted for control. Changes in the ore-to-coke ratio were made according to the blast temperature used. The blast temperature was 2475°F, for Test 11, 2600°F, for Test 12, and 2100°F for Test 13. Operations were smooth in all three tests.

The study of oxygen additions to the blast at the 2100°F level was performed in Test 15. As in Test 10, enough oxygen was added to the blast to burn the natural gas to CO and H_2. The coke per THM increased 15 lb compared to the test where no oxygen was used; however, hourly metal production increased by 90 lb over the same period. Table IV illustrates the effects of blast composition on coke rate and production rate.

Results

Although three runs were made at reduced wind rates to keep the bosh volume constant, most of the runs were made with a constant wind rate. Comparisons of results are made only for these latter runs. These operations have the advantage of being pertinent to actual plant operations. Normally, the wind rate would be held constant; therefore, any gas or moisture injections would be in addition to the normal blast.

Effect on temperature: Moisture added to the blast will consume coke in the tuyere regions as a result of the reaction between C and H_2O forming CO and H_2, and, since this reaction is endothermic, the moisture exerts a cooling effect in the tuyere region. If metal quality is to be maintained, this cooling effect must be offset by an appropriate increase in blast temperature. The relationship established between blast temperature and moisture content of the blast is shown in Fig. 6. The slope of this plot gives a required temperature increase of 34°F per grain of moisture per cu ft of blast, which agrees with the often-used value of 33°F per grain.

Natural gas injected into the blast also exhibits a cooling action; therefore, an increase in blast temperature is required to maintain metal quality. The

cooling effect results from the fact that the gas consumes oxygen which would otherwise burn coke, and that the heat liberated in burning the gas to CO and H_2 is much less than that for burning C to CO. Furthermore, the natural gas was not preheated. The relationship between percent natural gas added and blast temperature is given in Fig. 7. The slope of this plot corresponds to an increase of 140°F for each percent of natural gas injected (vol pct of blast air). This is somewhat higher than a predicted value of 110°F used as a guide in planning the tests.

Production rate: An increase in production rate was realized with both moisture and natural gas injection. The increase was possible because of the additional heat supplied in the blast. With the use of moisture, the higher production rate resulted from the faster driving rate and a higher burden ratio. With natural gas, increased production was the direct result of a much higher ore-to-coke ratio. The relationships between production rate and blast temperature are shown in Fig. 8. The increase in production for each 100°F increase in blast temperature was approx 40 lb per hr for both moisture and natural gas.

Coke rate: The major difference in behavior of moisture and natural gas is their effects on coke rate. Both permit savings in coke, but the savings with gas injection are much greater than with moisture injection. The respective savings were 50 lb and 13 lb per THM for each 100°F increase in blast temperature, as shown by the slopes of the appropriate plots in Fig. 9. This difference in coke savings is largely the consequence of the different manner in which the furnace must be burdened for equivalent additions of moisture and gas. Since moisture consumes coke, an increase in driving rate would be effected even without a change in burden ratio. Gas, on the other hand, consumes oxygen and will slow down furnace movement unless the ore-to-coke ratio is increased.

Injection of oxygen, equivalent to the natural gas, permitted a higher production rate at a given blast temperature than for gas alone. This result was expected, because the blast would consume coke at a greater rate and thus speed up furnace movement. The increase in production rate was in direct proportion to the amount of oxygen added to the blast, although no further drop in coke rate was achieved.

Hydrogen Utilization: When either natural gas or moisture is injected into the furnace, hydrogen gas is produced from the overall combustion reactions,

$$CH_4 + \tfrac{1}{2}\,O_2 = CO + 2H_2$$

$$H_2O + C = CO + H_2$$

Since the hydrogen should be a very active reductant toward the iron oxides in the furnace stock column, it would be expected that a portion of the hydrogen would be converted into water, in which form it would appear in the top gas. A plot of the hydrogen in the top gases vs hydrogen introduced through the tuyeres as moisture and as natural gas is shown in Fig. 10. The plot has been extrapolated to zero hydrogen input to find the hydrogen which would be expected from the burden. When this amount is deducted from the hydrogen in the top gas, the remainder represents hydrogen that was introduced with the blast. Fig. 11 shows that the percentage utilization was approx 45 pct. A value of about 50 pct was reported by Bonnaure at the 1959 Blast Furnace, Coke Oven, and Raw Materials Committee Meeting of AIME for a somewhat larger furnace.

Furnace control: Control of the furnace by variation of the natural gas content of the blast was not satisfactory. When the furnace was operating on the cold side, the rate of gas injection was decreased, as gas has a cooling effect. However, the reduction in gas freed a corresponding amount of blast oxygen for combustion of coke, and the driving rate of the furnace increased. The net result was a cooling effect, due to the delivery of more burden to the tuyere zone per unit time. Moisture variation was much more effective than gas in obtaining furnace control.

The successful utilization of natural gas in the experimental furnace has demonstrated the potential for gas injection on an industrial sale. In areas where the availability of cost of metallurgical coke is a limiting or a major economic factor, the injection of natural gas may prove to be of considerable value. Not only does the gas save coke, but it brings about an increase in furnace productivity. These benefits are realized without serious problems of metal quality, since the sulfur content of the gas is low and conceivably could be made even lower by appropriate gas-scrubbing operations. The operation of the furnace during the period of bosh injection of natural gas was unusual. The very high production rate is readily explained by the low utilization of the gas; although the burden ratio was increased, the carbon-consuming capacity of the blast was not correspondingly reduced. It is surprising that the furnace operated at the high blast temperature without hanging. Evidently the physical cooling effect of the gas and the rapidly moving stock column contributed to the smooth operation. Further work is indicated possibly a study of the effect of adding an inert gas, such as nitrogen, through the bosh tuyeres. These experiments also point toward the probable potential of injection of solid fuels, such as coal fines and char, which may be less expensive than the equivalent gas.

Conclusions

Based on the data obtained, it is concluded that:

1) Injection of natural gas into the main blast furnace tuyeres is technically feasible at elevated blast temperature; also the data suggests that natural gas injection could be used to effect at least small benefits through a wide range of blast temperatures;

2) Injection of natural gas by any of the three methods tried resulted in substantial coke savings and increased production rates;

3) The very high rate of production maintained while introducing gas in the bosh suggests the need for further research in this field;

4) Furnace control can be maintained by varying the moisture content of the blast; but gas variation is not effective;

5) Only a slight saving in coke, but a substantial increase in production rate, can be realized by the use of high blast-temperatures with suitable injection of moisture to control the hearth temperature; and

6) Oxygen can be added to a furnace blast enriched with natural gas to increase production, almost in direct proportion to the oxygen additions.

BLAST FURNACE INJECTIONS

This paper describes methods of calculation, which give the conditions to be fulfilled in order to tap the same quality of iron and have a smooth running furnace when a new material is injected into the blast. These calculations also show the effects of the injection upon the regularity of the iron, the production of the furnace, the coke rate, and the quality of gas produced. Included in the paper are the results of two industrial campaigns: one with natural gas and the other with light fuel oil.

by J. A. Cordier

A number of materials are available for blast-furnace injection, but industrially it is impossible to test all possibilities because blast-furnace experimentation is lengthy and costly, and the combinations of materials, techniques, and operating procedures are countless. However, theoretical considerations and simple calculations, based on reasonable assumptions, provide a means of selecting a limited number of solutions which should be worth investigating.

In testing the injection of different materials, it should be kept in mind that blast-furnace techniques and designs have been perfected over many years, and one should not greatly alter the working conditions of the existing, modern furnaces. The main conditions to be met are the following:

1) The thermal gradient in the stack should be maintained; hence, the heat capacity of the upgoing gases should be approximately equal to the heat capacity of the downcoming solids, as in any heat exchanger. This factor explains the advantages of lightening the burden by beneficiation and sintering. Fuel oil injected at the tuyeres also lightens the burden by reducing the quantity of coke charged which, in itself, represents a substantial saving.

If the gas volume is too large, the temperature gradient is shifted upward, resulting in a hot top. But if the gas volume is too small, the stack becomes cold, the iron oxides do not have a chance to be reduced before they melt, and the quality of the iron is degraded.

2) The temperature of the combustion zone in front of the tuyeres should be closely controlled. Behind the wall of coke, where most of the molten materials flow, very hot reducing gases (3600°F) heat up coke, slag, and iron to high temperatures which speed up the reaction:

$$(SiO_2) + C = CO + SiO \text{ (gas)}.$$

The silica comes from coke ash and slag; hence the difficulty of obtaining low-silicon iron and high blast-temperatures with high-ash coke or acid slags. The SiO can react with iron or primary (oxidized) slags by two mechanisms:

$$2(SiO) \text{ gas} = Si \text{ in iron} + (SiO_2) \text{ slag}$$
$$(SiO) \text{ gas} + (FeO) \text{ slag} = Fe + (SiO_2) \text{ slag}.$$

Through these two mechanisms, there is a tendency for SiO_2 to be retained in the bosh, much in the same way as ZnO is retained in the furnace at a higher level.

Dissolved in the primary slags, silica thickens them, and the paste makes a roof which protects the raceway. Under normal circumstances, this roof collapses at regular intervals, but if there is too much SiO, the SiO_2 builds up in the lower part of the bosh and clogs the furnace, the charge does not descend regularly, the furnace kicks, and the blast pressure increases. This situation can aggravate itself to the point of a serious hanging condition.

The well-known remedy is to cool the combustion zone with a colder blast or water vapor. The quality of coke and burden can have a great influence on the temperature, and injections into the blast also can provide the necessary control.

3) It is desirable to perform in the stack as much indirect reduction as is compatible with the reducibility of the ore and sinter. With lean burdens, there is always an excess of reducing gases in the stack because much coke is needed to satisfy the thermal needs of the charge. This is not always the case with very rich burdens where indirect reduction can be limited by the small amount of reducing gas per ton of iron oxides. Thus, it should be worthwhile improving the reducibility of lean sinters and increasing the quantity of reducing gases for very rich burdens. This can be done by the injection of hydrocarbons, compensated by a superheating of the blast or even an oxygen enrichment, if this proves economical.

Role of the stoves and gases

The stoves have a very high thermal efficiency with cheap fuels such as blast-furnace gas. The heat extracted from a gas at low calorific value is transferred at about 1450°F to the hot blast. By combustion with preheated coke, this same heat is boosted to 3700°F and transferred to a reducing gas. This marvelous heat pump insures the success of the blast furnace over the electric arc in melting at high temperatures under reducing conditions.

Since the stoves represent an important investment, and since they perform such a beneficial role, they should be used at the highest obtainable blast temperature; this can be done only with injections in the blast.

Nitrogen is necessary to transport heat from the stoves to solids in the stack. For lean burdens, there is not enough nitrogen in the air to perform this

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task. But for rich burdens, there is too much nitrogen, and it would be worthwhile to enrich the air with oxygen.

Carbon monoxide is plentiful when operating on lean burdens and not very well utilized, if we think that precious coke is burned to make it. Carbon monoxide is a good reducing agent at low temperatures, but it is not efficient at high temperatures where reactions with solids could proceed rapidly. In the bosh, carbon monoxide slows down the solution loss, but—triggered by the presence of reduced iron and hydrogen—it sustains carbon deposition in the temperature range of 800° to 1300°F.

Very fine carbon can be harmful, and the formation of carbon black from injected fuel oils and other hydrocarbons should be avoided. On the other hand, the formation of carbon black does not seem to be as harmful as deposited carbon.

Normally, hydrogen represents a small percentage of the gases in the blast furnace; it comes from the blast moisture, volatiles in the coke, and hydrates in large pieces of ore or fines. Hydrogen also can be injected as water vapor or hydrocarbons in the blast. It is a very good reducing agent, especially at high temperatures where it can compete with the carbon of the coke. Due to its low viscosity and its high diffusivity coefficient, hydrogen reaches the center of ore particles through minute microporosity faster than any other gases; thus, it precedes the carbon monoxide reduction front. The resulting H_2O diffuses back faster than CO_2 and transfers oxygen to CO at the surface.

The heat conductibility of hydrogen is from seven to ten times greater than that of other gases; thus, it accelerates the heat exchange between gases and solids, and narrows their temperature difference, thereby reducing the heat losses through the top gas. In the combustion zone, hydrogen helps in the heat transfer by means of *active convection*, i.e. the formation of free radicals—still not very well understood—which recombine at the surface of a solid to deliver very efficiently the energy absorbed at high temperatures.

This phenomenon has not been taken into account in the calculations; thus, the injection of hydrogen in one form or another, should permit the blast temperature to be increased to a higher level than has been calculated. This was observed for steam injections—from the theoretical 24.7° to 37°F per extra grain per cu ft—but unfortunately it was not observed for fuel oil and natural gas injections.

Injection principles

The role of gases in the blast furnace and the most efficient utilization of stoves, in addition to factors cited for the maintenance of blast-furnace working conditions, form the basic principles which we have used to find the correct and most economic conditions for the injection of materials into the blast; they can be summarized as follows:

1) the thermal gradient of the furnace should be maintained, and especially the temperature of the combustion zone,

2) the volume of the reducing gases should be adjusted to the needs of the indirect reduction of oxides,

3) the blast should always be at maximum temperature,

4) the formation of carbon black should be avoided,

5) it is always worthwhile to replace CO by H_2,

6) with rich sinters it is worthwhile to replace N_2 by CO or H_2, and

7) with lean, but reducible sinters, it is worthwhile trying to replace part of the CO by N_2 at a low driving rate.

Effects on regularity

The moisture content of the air can vary considerably. For the blast, it should be controlled, because a fluctuation of 1 grain per cu ft must be compensated by a change of blast temperature of at least 25°F to keep the combustion zone at the same temperature, and 35°F to keep the furnace hot when variation in moisture is to be permanent.

Blast moisture can be controlled by drying the air—which is quite expensive—or by injecting steam into the cold blast—which means that the moisture is always at the maximum level. Since steam is an expensive source of hydrogen and oxygen, we think that it should be kept at the lowest possible level in the blast, and replaced, if necessary, by some hydrocarbon injected in the blow pipes. A means of keeping moisture at a minimum and controlled level is by washing the compressed air in a cooling tower with cold water. If the blast is at 15 psi and the water at 60°F ± 10°, the moisture will be 3±1 grain per cu ft.

The effects of other uncontrolled fluctuations may have to be corrected. A change of the coke charge takes 10 hr to be effective and 6 hr with the ore, but a change in the blast temperature is effective immediately. While such a correction is quite applicable to small fluctuations, it is dangerous for larger changes, because the temperature of the combustion zone is dependent upon the temperature of the blast, and large increases in temperature of the combustion zone can provoke sticking and hanging as mentioned earlier. One solution to this problem is the following:

If the furnace is cold, the blast temperature can be increased, and nitrogen injected into the cold blast at the rate of 4.47 pct of the air volume for 100°F. The total heat input per ton of coke burned at the tuyeres is double the value without nitrogen. The thermal gradient in the furnace is not affected. When blowing at constant pressure or pressure drop, less coke is burned, and the furnace slows down, which helps in warming it up.

When injecting some material steadily into the blast, it is an easy matter to control immediately small fluctuations of the furnace without touching the blast temperature, which should be maintained at a maximum. This control can be accomplished by adjusting the quantity of material injected. However, if the fluctuations are large, nitrogen should be injected until the coke correction is effective.

Effects on production

The production of a blast furnace can be referred to as P tpd per 1000 cu ft of working volume. This term has a physical meaning when the gases are reasonably distributed over the cross section of the furnace.

When the driving rate is increased, the coke rate goes up slowly, because there is a reserve zone in

the stack where exchanges of heat and oxygen between gases and solid come to a stop. Thus, there was too much furnace volume per ton of iron, and the driving rate was too slow. If the furnace is blown very hard, there is no reserve zone left, and indirect reduction is curtailed, causing a sharp increase in the coke rate. A partial fluidization of the solid bed occurs; the gases in large quantities go up chimneys without meeting any solids; and the coke rate and the quality of the iron are disastrously poor.

A good working furnace should be operated just below this state. When the commercial situation requires more iron, one solution is to beneficiate the burden or charge scrap. Injections enable production, which was limited by stack reactions, to be increased for the following reasons:

1) Some of the coke is replaced by another fuel injected at the tuyeres. The weight of this coke can be replaced in the charge by a thermally equivalent weight of ore which will not change the retention time of solids or gases.

2) If the material injected into the furnace is hydrogen-bearing, or increases the CO + H_2 percentage in the combustion gases, the kinetics of reduction and heat transfer will be enhanced, and it will be possible to drive the furnace faster, while keeping the same performance. Different materials can be ranked in terms of the pct of extra hydrogen (h) that they introduce into furnace gases. For a superheating of the blast of 180°F, pct h is equal to: 3.8 for cold hydrogen, 2.35 for cold coke-oven gas, 2.15 for natural gas, 2.0 for fuel oil atomized with coke-oven gas, 1.9 for rest gas, 1.75 for fuel oil atomized with compressed air, 1.35 for high-volatile coal, and 1.30 for steam. It can readily be seen that steam is not a good source of hydrogen; coke-oven gas or natural gas is preferable.

If the presence of hydrogen improves blast-furnace operations by increased indirect reduction, lower heat losses in top gas, and better heat transfer in the bosh, then the overall heat and reducing gas requirements per ton of iron can be reduced considerably.

If we assume that the weight of burden plus coke is constant, we imply that heat requirements are constant per unit of weight. If this is not the case, then we should keep constant the retention time of the gases, or if the void index of the burden is the same, the gas production of the furnace should be kept constant. And if the total heat balance of the furnace per ton of iron has dropped by pct H_2, then we can increase production by pct H_2. The overall production increase is,

$$\frac{100\ (-\Delta C)}{C + 1900\ Fe/100} + a\ \%h + \%H = \frac{\Delta P}{P} \times 100$$

where $1 < a < 5$.

Experience with fuel oil and natural gas has shown that it is economical to work at constant coke consumption. If it is necessary to go beyond the limit of the stoves, oxygen enrichment of the blast and a further increase in the quantity of material injected, could be used.

Our conclusions are valid for overblown furnaces, for their characteristics can be brought back to normal with more air blown or by applying high top-pressure techniques. With fuel or gas injections it should be possible to have the same production as before, but with a good coke rate and a smoothly operating furnace.

Hydrocarbon injections are a competitor to high top-pressure techniques. They can be combined to obtain really high-production rates with good coke rates and good quality iron which never could be reached with injections alone, because beyond a certain point, there is fluidization of the charge which only high top-pressure can stop.

In addition to fluidization, there is another phenomenon which can limit the production rate, especially with sinters containing less than 50 pct Fe; it is the reducibility of the oxides.

Charging hot sinter can increase considerably the production rate of the furnaces with injections because it makes good use of the reducing gases injected into the furnace. It also has other advantages:

1) The carbon deposition reaction cannot take place because the iron oxides, which catalyze the reaction at about 1000°F, are too hot; this avoids many problems, especially with hydrocarbon injections, since the presence of hydrogen strongly accelerates carbon deposition. The reactions in question can be written (in the order of increasing speed):

$$CO + CO \rightarrow CO_2 + C$$
$$2CO + CaO \rightarrow CaCO_3 + C$$
$$CO + H_2 \rightarrow C + H_2O$$

2) With high top-pressure and self-fluxing sinter, the free CaO and the calcium ferrites can react with CO_2 between 1000° and 1600°F according to the following reactions:

$$CO_2 + CaO = CaCO_3$$
$$CO_2 + CaOFe_2O_3 = CaCO_3 + Fe_2O_3$$
$$CO + CaOFe_2O_3 = CaCO_3 + 2FeO$$

The carbonate, thus formed, is dissociated at 1670°F (and up to 1800°F according to the pressure) in a lower part of the furnace. There is an accumulation of carbon dioxide between the 1000° and 1670°F isotherms, both in the solids going down and the gases going up. This carbon dioxide poisons the gas, burns some carbon of the coke, and takes up useful heat at 1670°F to deliver it at 1000°F where it is wasted. There is good practical evidence of this phenomenon, which can be avoided by charging hot sinter.

3) The rapid and abrupt cooling of the sinter, even in large air coolers, stresses the glassy material which binds the crystals and makes it more fragile. Hot sinter is still pasty and does not make as many fines.

Slow driving rate

During periods when pig iron is in slack demand, it may be interesting to put blast furnaces on a reduced blast. The injection of nitrogen into the cold blast can be very worthwhile under such circumstances, provided nitrogen is available at a reasonable price.

At low driving rates, the gases and solids stay in the furnace much longer, and they are almost in equilibrium with the charge. The quantity of reducing gases per ton of iron can be reduced without affecting the indirect reduction, and it is possible to inject nitrogen into the cold blast and raise the blast temperature or replace a hydrocarbon at the tuyeres with nitrogen in the cold blast. The savings can be very interesting.

Table I. Variation of Blast Furnace Driving Rate by Injections

	Slow	Reference	Normal	Fast	Accelerated
Material injected	N/3	—	Fuel	Fuel	Fuel + O_2
Blast temp, °F	1875	1475	1875	1875	1475
O_2 in blast, pct	17	21	21	21	27
N_2 or O_2, cu ft/THM	11,500	0	0	0	5,100
Fuel, lb/THM	0	0	154	172	310
Coke, lb/THM	1210	1540	1080	1270	1400
Coke saving, lb/THM	330	—	460	270	140
pct	21.5	—	30.0	17.5	18.2
Top temp, °F	300	400	300	400	480
CO_2/CO	0.625	0.365	0.610	0.354	0.268
Indirect reduction, pct	73	65	80	85	65
Top gas, Btu/cu ft	71.5	99	81.5	106	135
Top gas, Btu x 10^6/THM	6.2	9.4	6.95	9.7	12.9

Table II. Pct CO + H_2 in Combustion Gases According to Material Injected, Reference 36.2 Pct

Material Injected	H_2	H_2O	Coke-Oven Gas	CH_4	Fuel Oil
Blast Temp + 400°F	42	40.1	40.1	39.4	39.0
Oxygen at 27 pct	—	50.9	49.5	48.7	48.0

An example of the adaptation of the blowing and fuel conditions to production requirements has been calculated and is shown in Table I. This table refers to a burden of 100 pct self-fluxing sinter, made of crushed Minette ore of 37 pct Fe. Analysis of the hot metal is as follows: 3.5 pct C, 0.5 pct Si, 0.4 pct Mn, and 1.9 pct P.

Effect on coke rate

Carbon charged into the furnace, per ton of iron, goes:

in the hot metal		70 lb
in the reduction of		
SiO_2, MnO, P_2O_5, and FeS		30 to 70 lb
in the flue dust		10 to 40 lb
		110 to 180 lb
The remainder is burned, either by		
CO_2 or H_2O in solution loss	d_1 lb	D lb
FeO in direct reduction	d_2 lb	
the blast at the tuyeres		T lb.

Thus, to decrease the coke rate, there are several possibilities:

1) replace part of carbon, T, with some other fuel at the tuyeres,
2) decrease D, i.e. improve indirect reduction, or
3) decrease T by decreasing the thermal requirements per ton of hot metal (TMH).

1) Substitution of another fuel for coke at the tuyeres: The temperature of the combustion zone should remain constant, and this factor can be used to write a quantitative relation between the amount of material to be injected per unit volume of blast and the superheating or oxygen enrichment of the blast necessary to compensate for the cooling effect of the injection.

These calculations consist of making a material and heat balance of the combustion zone for the reference period and for the injection period, assuming that the temperature of the combustion gases (CO, H_2, and N_2) are the same in both conditions. The material balance is referred to 1000 Btu transported as sensible heat by the gases. The overall calculation of the benefits to be derived from the injection can be made by assuming that the thermal requirements of the charge have not been changed.

Materials injected can be ranked in the order of their replacement value for coke. For 1 lb of coke, it takes (with superheated blast): 1.11 lb of coal, 0.775 lb of fuel oil, 15.2 cu ft of CH_4, 20.8 cu ft of rest gas, 37.7 cu ft of coke-oven gas, and 77 cu ft of H_2.

With oxygen enrichment of the blast, the results are rather poor for this aspect of the question, because the blast, containing less nitrogen, brings less heat from the stoves per unit of carbon burned at the tuyeres. Thus, there is a deficit which must be compensated by burning more fuel.

2) Improve indirect reduction: D, the weight of carbon burned by direct reduction and solution loss can be decreased by proper injections. If the sinter is poor in iron, there is plenty of carbon monoxide, but it may not be well utilized because lean sinter is not readily reducible. Hydrogen, as mentioned previously, catalyzes the reduction reactions. At high temperatures, hydrogen also performs reduction on molten primary slags which carbon monoxide would have left to coke carbon. Both phenomena improve the indirect reduction and decrease the weight of carbon, D.

Different tests, especially thorough stack investigations by probes made by E. P. Bonnaure at the International Low Shaft Blast Furnace at Liège, Belgium, under varied conditions have shown that 35 to 50 pct of the injected hydrogen was used to improve the indirect reduction—most of the values centered around 40 pct.

It can be calculated that, for an increase of 1 pct H_2 in the combustion gases, the indirect reduction coefficient (and D) is improved by 3 pct with a 100-pct self-fluxing sinter burden and 4.5 pct with a 100-pct unprepared burden. It is, thus, worthwhile injecting high-hydrogen materials into blast furnaces where the top gases are of high Btu content and the indirect reduction coefficient low. Coke-oven gas is well suited, and steam gives the smallest increase of hydrogen in the combustion gases.

If the sinter is very high in iron, it may be that, even though the top gases are low in Btu content, the indirect reduction coefficient is low because there is not enough reducing gas to satisfy the needs of the reducible sinter. In this case, combustion gases should be enriched in carbon monoxide and hydrogen, even with oxygen enrichment of the blast, if this proves to be economical. Table II compares different materials in this respect; it gives the pct (CO + H_2) in the combustion gases obtained with a superheating of the blast of 400°F or an oxygen enrichment of 6 pct.

3) Decrease thermal requirements per THM: The most efficient way of reducing the coke rate is to reduce heat requirements per THM, since they account for about 80 pct of the coke consumption. Im-

provement of the indirect reduction coefficient can reduce considerably the heat requirements per THM. Hydrogen can also be a factor in reduced thermal requirements, since its injection improves heat transfer and promotes an increased production which lowers heat losses and, as a consequence, the coke rate.

Calculations

Reference conditions of the blast furnace are first analyzed. Material and heat balances are calculated for the combustion zone and the rest of the furnace. The conditions at the tuyeres referred to 1000 Btu are then calculated for several materials which are likely to give good results and for the maximum superheating of the blast possible with the stoves. Oxygen enrichment can also be considered. The new top temperature, indirect reduction coefficient, and heat losses are estimated from the anticipated working conditions of the furnace, past experiences with injections, and the analysis of the combustion gases in H_2 and $(CO + H_2)$. Several estimates can be used so as to be able to judge which one is the most realistic.

The heat and material balance is calculated for the new conditions. From the total heat requirements and the conditions at the tuyeres per 1000 Btu, it is possible to calculate the blast volume and the total quantity of material to be injected. And, with the indirect reduction coefficient and the analysis of the oxides, the top gas volume and composition are computed.

Many sets of calculations were made. The general conclusions are as follows:

1) If coke is expensive, materials with high $C:H_2$ ratios should be injected with very high blast-temperatures. The driving rate should be kept at a normal level.

2) If a high production rate is required, materials with high $H_2:C$ ratios should be injected with high blast-temperatures and even with oxygen enrichment if necessary.

3) With very rich sinter burdens, oxygen enrichment and injection of high $C:H_2$ materials should be worthwhile, provided the price of oxygen is not too high. If this is not the case, H_2O, CO_2, or a high $H_2:C$ material could be injected with an increase in the blast temperature.

4) With lean sinter burdens, at slow driving rates, the injection of nitrogen in the cold blast can be economical, if nitrogen is available at a very low price.

Technological difficulties

The injection of steam, carbon dioxide, flue gases, nitrogen, and oxygen into the cold blast is no problem. But the injection of reducing materials presents more difficulties. A proposed solution is to introduce the material as far as possible into the tuyeres so as to prevent premature combustion that might damage the blow pipe or tuyere. But there is a risk of forming carbon black, which would not burn and could cause problems in the stack.

A second solution is to make a combustion chamber outside of the blast furnace and partially burn the reducing material until there is no free carbon left. But this leads to a slightly oxidizing gas at a very high temperature which is difficult to handle. Furthermore, the device is cumbersome, and the heat losses are high.

To inject atomized light fuel oil or natural gas, we found it more convenient to place the injector along the axis of the blow pipe at a distance from the end of the tuyere equal to about four times its diameter. In that way, the outer surface of the flame does not touch the inner wall of the tuyere, and there is enough precombustion to avoid the formation of carbon black. The fuel or gas flame should be inside of the coke flame, or in other words the raceway. Cracking should be avoided in the injection tube in the axis of the blow pipe. Compressed gas circulates at high enough speed inside of the small-diameter stainless steel tube so as to keep the inner wall temperature below the cracking temperature.

When injecting fuel oil, the atomizing air is circulated around the fuel tube and inside of a stainless steel tube which itself is protected against convection and radiation by an outer thin tube of high-alloy steel. Thus, the quantity of atomizing air can be kept below 0.3 lb per lb of fuel. This is important because compressed air is expensive and cools the blast; with 0.5 lb of air per lb of fuel, 8 pct of the superheating of the hot blast is absorbed by the cold compressed air. Compressed air can advantageously be replaced by natural gas or coke-oven gas.

Safety devices should be installed that cut off gas or fuel, if the bustle pipe pressure, or if the air flow in one single tuyere drops to a preset level. It would indeed be dangerous to continue injecting oil or gas into a blow pipe with the tuyere plugged by slag, for the gas would burn in the bustle pipe and probably damage it.

Since hydrogen is very efficient at high temperatures, it is preferable to inject it as low as possible in the furnace. It has also more chance to react with the solids and to be well mixed with the main stream of gases and be well distributed over the cross section of the furnace, if it is injected through the normal tuyeres.

Nevertheless, it may be interesting to inject some material through another set of tuyeres, higher in the furnace, in the bosh for instance, to help a furnace with a worn-out lining move down more evenly. By this technique, a larger quantity of material can be injected, since the temperatures at bosh level are much lower than at the tuyeres. In addition, a top gas of high calorific value can be produced.

Industrial test with fuel oil

Fuel-oil injection tests, performed on blast furnace no. 3 of the Forges et Acieries de Pompey, were sponsored by the European Coal and Steel Community.

The blast furnace has eight tuyeres and burns 270 short tons of coke per day. The burden consists of a mixture of unscreened acid and basic Minette ore, under 10 in. in size. Metallic iron additions were kept at a minimum so that the results would be more significant. Average analysis of the hot metal was: 0.5 pct Si, 0.4 pct Mn, 1.8 pct P, 3.5 pct C, and 0.040 pct S.

The first part of the test consisted of a two months' reference period and eight periods of one week, during which several techniques were tested: injections of fuel oil in the bosh and in the normal tuyeres through two, four, and then eight points, with and without utilizing bosh tuyeres. Low and high sulfur fuels were used; the fuel rate range ex-

plored was from 60 to 160 lb per THM. Finally, three techniques were selected, and each was tested over a one month period.

First period: Light fuel oil atomized with compressed air was injected along the axis of the blow pipes of the eight normal tuyeres at the rate of 90 lb of oil per THM. To compensate for the cooling effect of the fuel and the cold compressed air, the blast temperature was raised from 1450° to 1625°F. In order to tap the same quality iron, the charge was increased until the coke rate had dropped by 205 lb or 10 pct. The furnace was blown so that the flue dust production remained constant at 400 lb per THM. Under these conditions, the coke charge was maintained and iron production went up by 10 pct.

Second period: The furnace was driven under the same conditions as during the first period, except that two of the four bosh tuyeres were put into service. The fuel rate did not change, but the coke rate increased by 60 lb. when the coke charge was increased the result was an extra 8 pct gain in production—18 pct in all. The flue dust per THM went up to 500 lb.

Third period: The injection of atomized fuel oil was made in the blow pipes of the four bosh tuyeres in service. The fuel rate was kept at the same level, but, for the same increase in production (18 pct), the coke rate increased by 40 lb. The operation was not as satisfactory as the previous tests.

The quantity of fuel oil injected per THM corresponded closely to the figure calculated by the method described. But the superheating of the air had to be corrected because of the larger quantity of air used, as compared with the 0.5 ratio used in the calculation.

The working conditions of the furnace improved markedly. There was an increase of the metal temperature of 50°F, and the sulfur in the iron dropped by about 40 pct, with the sulfur partition coefficient changing from 25 to 40.

Industrial test with natural gas

Natural gas injection tests were performed on blast no. 3 at the LeBoucau works of the Compagnie des Ateliers et Forges de la Loire. These tests were sponsored by the Direction de la Siderurgie Francaise and Gaz de France.

The blast furnace has six tuyeres and burns 155 tons of coke per day. The burden is made up of a mixture of several unscreened silicious hematite ores of high iron content and limestone. The hot metal (foundry iron) had the following analysis: 2.5 pct Si, 0.8 pct Mn, 0.08 pct P, and 4.0 pct C. The slag weight was 800 lb per THM and had a basicity index of 1.2.

Natural gas from Lacq (97 pct CH_4) was injected at 125 psi along the axis of the six blow pipes. The blast temperature was raised step by step during two weeks from 1445° to 1870°F. At the same time, the gas flow rate was gradually increased to 22,000 cu ft per hr. Calculations had shown that 8 cu ft of gas should be injected per 1000 cu ft of hot air for every 100°F of superheating of the blast. This rule was followed, and experience showed that a slight deviation from it produced either hanging or cold iron of low silicon content.

When the furnace was stabilized at 1870°F blast temperature, the coke rate had been reduced by 16.6 pct or 296 lb of dry coke per THM with an injection of 2880 cu ft of natural gas per THM. Iron production went up by 14.6 pct and was limited only by the capacity of the charging equipment. Utilization of the injected hydrogen as a reducing agent avgd 40 pct, whereas carbon monoxide utilization was less than 20 pct.

The top gas flow rate was kept at the same level as with previous practice, while the hydrogen content of the top gases increased to 6 pct from 2.1 pct. The high momentum of the natural gas changed the mechanical conditions of the raceway. Investigations with probes in the stack at the beginning of the test showed that the gas flow was uneven and central. The tuyeres were shortened by 2 in., and gas distribution came back to normal, as was shown by probing the gases in the stack along two perpendicular diameters.

Conclusions

The thermal gradient in the furnace, and especially the temperature of the combustion zone in front of the tuyeres, should be kept as they were when the furnace was operating satisfactorily without injections. Superheating or oxygen enrichment of the blast to compensate for the cooling effect of the injection can be calculated by a thermal balance of the combustion. At all times, there should be sufficient reducing gases in the stack to achieve the indirect reduction compatible with the reducibility of the burden and the driving rate.

A simplified method of calculation, based on the knowledge of the normal performance of the furnaces and on two simple assumptions, which can be checked, gives the performances of the furnace with injection. The accuracy is sufficient to plan an injection test program.

Results of tests performed at the International Low Shaft Blast Furnace at Liege and industrial-size furnaces at Pompey and Le Boucau in France with fuel oil, natural gas, rest gas, steam, oxygen enrichment, and superheating of the blast have substantiated the assumptions made and confirmed, with a rather high degree of accuracy, the results of the calculations.

The results have shown that:

1) Only fast driving rates on furnaces charged with rich sinter can justify the use of oxygen enrichment with the injection of hydrogen-bearing materials;

2) Blast temperature should be as high as the stoves permit;

3) Materials high in hydrogen should be used when the driving rate is fast as is the case when the burden is not prepared;

4) Materials high in carbon with high temperature blast are adapted to normal production rates;

5) For slow driving rates, nitrogen can profitably be injected into the cold blast;

6) Nitrogen injection into the cold blast can be used as an efficient means of thermal regulation of the furnace.

If the new techniques and materials, as well as the important investment put into charge preparation, are to be exploited to the maximum, new blast furnaces should be designed for 1) high production rates—125 short tons per 1000 cu ft of working volume per day—2) charging with hot self-fluxing sinter containing more than 55 pct Fe; 3) blowing with air at 2100°F with hydrocarbon injection into the tuyeres and 4) a top pressure high enough to prevent fluidization—60 to 70 psi.

INJECTION TECHNIQUE INVESTIGATED ON LOW-SHAFT FURNACE

Investigations on various injection techniques at the international experimental blast furnace at Liège, Belgium, are reported, and a discussion of their influence is presented.

by E. P. Bonnaure and M. L. Halbrecq

The first tests on fuel-oil injections into the tuyeres of the International Low-Shaft Blast Furnace at Liège, Belgium, were performed in 1958. The results were presented at the 1959 conference of the AIME Blast Furnace, Coke Oven, and Raw Materials Committee meeting in St. Louis.

Only every other tuyere was fitted with a fuel-oil injector, but in spite of the uneven repartition of the fuel, the results were interesting enough to justify an investigation with fuel-oil injection into every tuyere with superheating or oxygen enrichment of the blast with and without high top pressure. The program also included injection of reducing gas containing 60 pct methane. Conditions of the injections were calculated according to the methods presented by J. Cordier in this issue of JOURNAL OF METALS.

To avoid preheating, the fuel-injection layout which used the light fuel-oil with approx the following composition: 85 pct C, 12.8 pct H_2, and 1.5 pct S, with 900 Btu per lb—heat of cracking—was pumped at 100 psi from a tank through a filter into the system. The flow was measured and controlled with an integrating flow-meter and 10 rotometers —one for each tuyere. Atomizing was accomplished with compressed air in the ratio of 0.8 lb per lb of oil, and this air also served as a cooling agent in the injectors. Although the atomization of oil was satisfactory, it is believed that, with improved injectors the necessary quantity of air could be reduced. Figs. 1 and 2 show the design of the injectors and their position in the blow-pipe. Flow rates up to 6 gal per hr per tuyere were attained with this set-up.

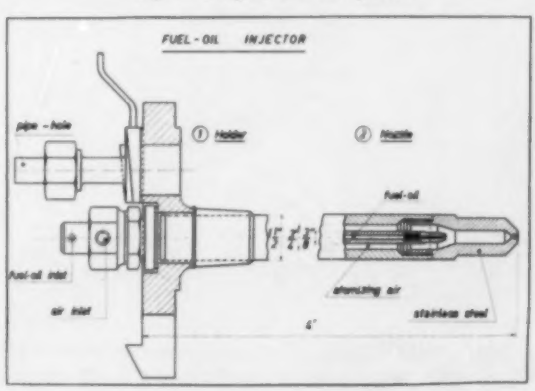
Injection of light fuel-oil with superheating of the blast

A coke-ore charging sequence was employed during most of the campaigns. The charge consisted of 880 lb of B nut coke, ⅜ to 1 in. in size, and a 100-pct self-fluxing sinter, ⅛ to 1 in., in size. Limestone was not charged, despite the low basicity index ($CaO/SiO_2 = 1.0$) of the slag. To insure more even distribution of the gases over the cross-section of the furnace and to decrease the pressure drop through the furnace, coke and sinter were charged in mixed form during several campaigns. The stock-line was maintained between 13 and 14 ft above the tuyere level. Thomas (basic bessemer) iron with the following analysis was produced: 0.30 to 0.80 pct Si, 0.30 to 0.50 pct Mn, 1.60 to 1.90 pct P. Table I provides a summary of the operation.

The high humidity and ash content of the coke required the conversion of actual coke rates into *normalized* coke rates, expressed in pounds of coke at 3 pct moisture and 10 pct ash. The arbitrary character of these corrections is recognized, but they are necessary for easier comparison with plant-scale blast furnaces. Assumptions used in the conversion are: 1) 1 pct moisture increases the coke rate by 1 pct, 2) 1 pct ash increases the coke rate by 1.5 pct, and —in order to convert the data to constant silicon in the iron—3) 0.1 pct Si raises the coke rate by 1 pct.

Tests at low and high top pressure with and without fuel-oil were run, and a constant bosh gas flow rate—approx 170,000 cu ft—was maintained to demonstrate the influence of the reducing power of the gases enriched in hydrogen by the fuel-oil

Fig. 1—Design of fuel-oil injector.



E. P. BONNAURE (IRSID) and M. L. HALBRECQ (Cockerill-Ougrée) are superintendent at the International Low-Shaft Blast Furnace at Liège, Belgium, and president of the Executive Committee for the undertaking, respectively.

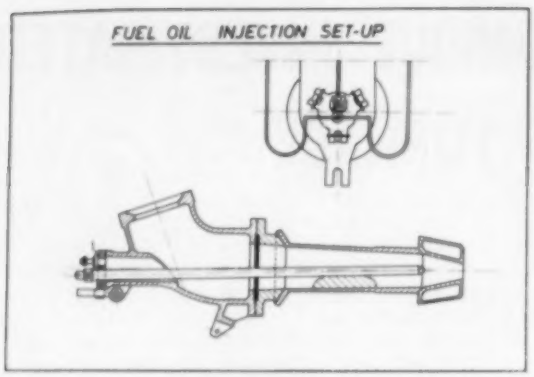


Fig. 2—Fuel-oil injection set up.

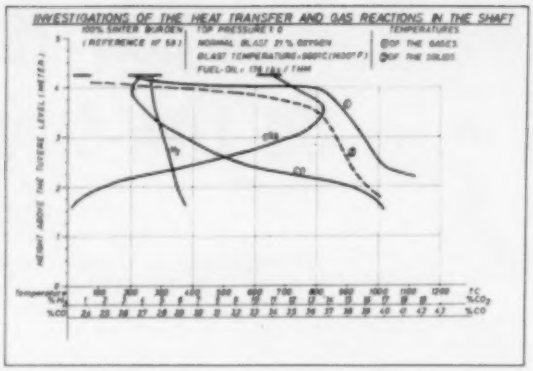


Fig. 4—Shaft reactions with fuel-oil injection at zero top pressure.

injection. In all cases, the furnace operation was smooth with no carbon black detected at the top.

Table I indicates the coke savings attained with the injection method. About 1/3 of the hydrogen is consumed in the reduction. Fuel oil notably improves the indirect reduction, and the increased hydrogen content of the top gas maintains its calorific value at the same level. Hydrogen appears to be used simultaneously as a reducing agent and a catalyzer of the reduction by CO according to the cycle:

$$MO + H_2 \rightleftarrows H_2O + M$$

$$H_2O + CO \rightleftarrows CO_2 + H_2.$$

Temperature measurements and gas analyses performed during the tests are illustrated in Figs. 3 through 7. At top high pressures—Figs. 5 and 6—a *reserve zone* occurs where the CO and CO_2 curves are parallel; therefore, this is a zone in which essentially no reactions take place. Fig. 6 shows that hydrogen is active as a reducing agent in the high temperature zone, and that its utilization is smaller at lower temperatures where CO becomes more effective.

Table I. Effect of Fuel Oil Injection with Superheating of the Blast

Burden
100 pct self-fluxing sinter: basicity ratio 1.25, size ⅛-1 in.
B nut coke: 13-15 pct moisture, 10-13 pct ash
Slag
Basicity ratio, $CaO:SiO_2$, 1.0
Vol, 1500 lb/THM

Top pressure, psi	0		30	
Fuel oil, lb/THM	0	176	0	160
Blast temp, °F	1470	1650	1470	1650
Production				
THM/day/cu m working vol	1.9	2.2	2.2	2.5
Top gas				
CO/CO_2	3.1	2.5	2.2	1.9
H_2, pct	1.8	4.2	1.7	3.7
Temp, °F	450	410	315	350
Btu/cu ft	108	105	97	96
Coke rate				
lb nut coke/THM	2040	1550	1780	1400
normalized rate	1680	1330	1460	1210
Iron analysis				
Si, pct	1.26	0.66	0.89	0.73
S, pct	0.050	0.084	0.041	0.079
Eff H_2 reduction, pct	—	30	—	40
Indirect reduction, pct	52	58	59	65

Injection of light fuel-oil with oxygen enrichment of the blast

Similar operating procedures were employed in the tests to determine the effects of oxygen enrichment. A summary of the operations and the results are given in Table II and Fig. 7. Difficulties encountered in this series are due to the incomplete combustion of the fuel-oil as evidenced by the appearance of carbon black at the top. A possible explanation is that the enrichment of the blast in oxygen increases the combustion temperature and decreases the volume of the oxidizing zone in front of the tuyeres. To maintain the same thermal level of the bosh gases, fuel injection must be increased, but because of the technical limitations of the in-

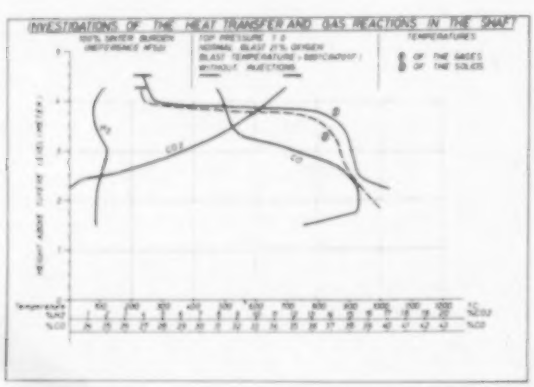


Fig. 3—Shaft reactions without injection at zero top pressure.

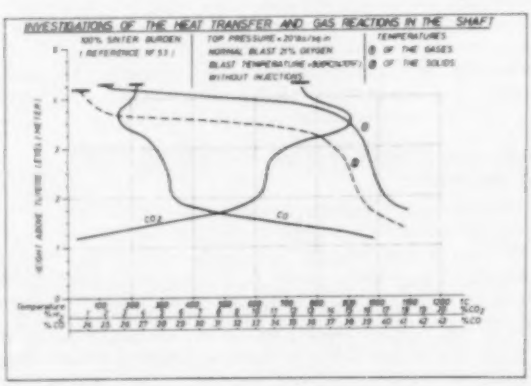


Fig. 5—Shaft reactions wihout injection at high top pressure.

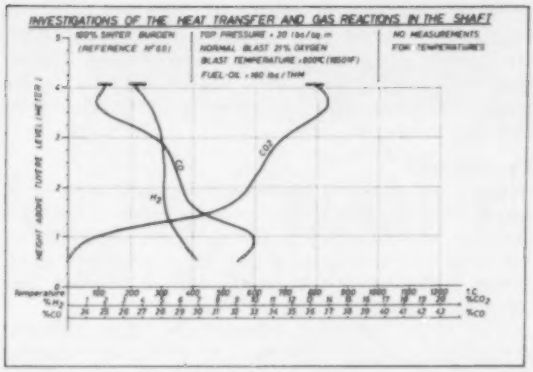

INVESTIGATIONS OF THE HEAT TRANSFER AND GAS REACTIONS IN THE SHAFT
100% SINTER BURDEN
(REFERENCE N° 65)
TOP PRESSURE = 20 lbs/sq in
NORMAL BLAST 21% OXYGEN
BLAST TEMPERATURE = 800°C (1850°F)
FUEL-OIL = 180 lbs/THM
NO MEASUREMENTS FOR TEMPERATURES
HEIGHT ABOVE TUYERE LEVEL (METER)
CO
CO2
H2

Fig. 6—Shaft reactions with fuel-oil injection at high top pressure.

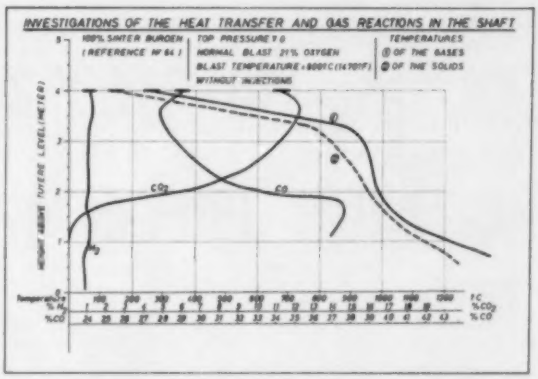


Fig. 8—Shaft reactions without injection at zero top pressure.

Table II. Effect of Fuel Oil Injection with Oxygen Enrichment of the Blast

Burden / Slag	See Table I.			
Top pressure, psi	0		20	
O_2 in blast, pct	21	24	21	24
Blast temp, °F	1470	1470	1470	1600
Fuel oil, lb/THM	0	266	0	180
Production				
THM/day/cu m working vol	1.9	2.5	2.2	3.5
Top gas				
CO/CO_2	3.1	2.1	2.2	2.1
H_2, pct	1.8	5.5	1.7	4.3
Temp, °F	450	340	315	235
Btu/cu ft	108	111	97	108
Coke rate				
lb nut coke/THM	2040	1310	1780	1300
normalized rate	1680	1100	1460	1150
Iron analysis				
Si, pct	1.26	0.66	0.89	0.25
S, pct	0.050	0.078	0.041	0.194
Eff H_2 reduction, pct	—	38	—	40
Indirect reduction, pct	52	65	59	61

jectors used, the fuel is projected past the short oxidizing zone, and incomplete combustion results. Because of this, tests will be resumed with a new type of injector for which better results are expected. Table II illustrates that, even at low pressures, the use of fuel-oil and oxygen allows high production with a low coke rate, produces a quality iron, and attains a reasonable calorific value of the top gas.

Rest-Gas injection with superheating of the blast

Table III summarizes the tests performed with a 60 pct CH, *rest-gas*. These tests were performed with L nut coke; therefore, they are not comparable with the previous tests which employed B nut coke. The layout was similar to the one used in the previous tests, although it was less complicated because of the elimination of the atomizing steps necessary in fuel-oil injection. For the same reason, a simpler injector arrangement was employed.

At normal pressure the injection of superheated rest-gas increases the indirect reduction from 54 to 60 pct, and decreases the coke rate by 13 pct for the same iron production. It should be noted, however, that during the campaign with the superheated rest-gas, the weather was extremely cold and the moisture content of the air was 60 pct lower; this is equivalent to a blast temperature increase of 80°F. At high top pressures, the superheated rest-gas injection increases the driving rate, and the production of the furnace increases by approx 50 pct for similar iron quality, indirect reduction, and coke rates.

In these tests none of the difficulties noted with fuel-oil were encountered. In all of the tests involving rest-gas, the Btu content of the gas was kept at a constant level, due to the approx 60 pct H_2 left over in the top pressures. As in the fuel-oil tests, a small reserve zone occurred, and hydrogen was used efficiently as a reducing agent at temperatures greater than 1800°F (see Figs 8 and 9).

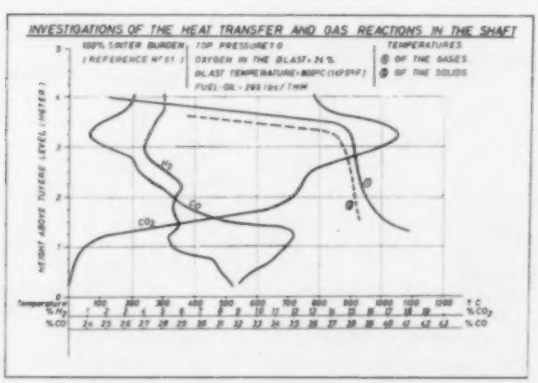


Fig. 7—Shaft reactions with fuel-oil injection with O_2 enrichment at zero top pressure.

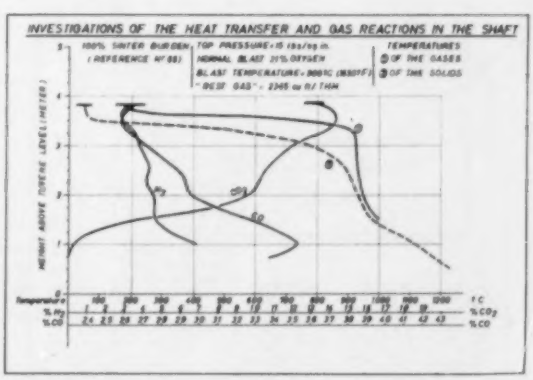


Fig. 9—Shaft reactions with rest-gas injection at high top pressure.

Table III. Effect of Rest-Gas Injection with Superheating of the Blast

Burden			
100 pct self-fluxing sinter: $CaO:SiO_2$ 1.25 size, size ¼-1 in. ⅛—in. size, 16-19 pct moisture, 10-12 pct ash			
L nut coke: ½-1 in. size,			
Rest-gas, 60 pct CH_4, 14 pct N_2, 13 pct Co_2, 5 pct H_2			
Slag			
Basicity ratio, $CaO:SiO_2$ 1.0 [1]			
Vol, 1500 lb/THM			
Top pressure, psi	0	0	14
Blast temp, °F	1470	1650	1650
Rest-gas, cu ft/THM	0	2900	2365
Wet coke consumption, tpd	44	33.5	48.5
Production			
THM/day cu m working vol	2.2	2.2	3.2
Top gas			
CO/CO_2	2.8	2.4	2.3
H_2, pct	1.1	3.9	3.6
Temp, °F	300	300	285
Calorific value BTU/cu ft	102	101	101
Coke rate			
lb nut coke/THM	1840	1300	1510
normalized rate	1480	1280	1300
Iron analysis			
Si, pct	0.81	0.53	0.38
S, pct	0.076	0.055	0.087
Eff H_2 reduction, pct	—	34	40
Indirect reduction, pct	54	60	60

Coke effects

Definite influence of the coke origin on the performance of the low-shaft furnace was observed during these tests, even though the chemical properties were similar and the granulometries were identical. It was thought that the low-shaft furnace was not influenced by the mechanical properties of different cokes, because the furnace height is about 1/5 that of the conventional furnace, and solid materials are retained only half as long. But since the low-shaft furnace runs smoothly with a 100-pct sinter burden, factors normally of secondary importance are brought into evidence. Previously, these factors could not be evaluated with good accuracy. However, the corrected coke rate with B nut coke is about 1680 lb, while it is 1480 lb with L coke. This amounts to a significant difference of some 7 pct, which is probably due to the differing natures of the coke.

These differences are attenuated by injections and even more so by high top pressure, which leads to the belief that the performances of the low-shaft blast furnace are influenced to a greater extent by high top pressure and hydrogen injections. Thus, the quality of the coke appears, under these circumstances, to be of secondary importance. This opens a new field of investigations on coke quality at the low-shaft furnace.

Conclusions

Since the purpose of the investigation was to develop and evaluate injection techniques in the blast furnace, the influence of the different variables was not explored systematically and no correlations between difference parameters can be derived from the results of this oriented research.

The tests, however, have shown the following advantages of the injection of reducing materials into the tuyeres with superheating or oxygen enrichment of the blast:

1) from 35 to 50 pct of the injected hydrogen is used as a reducing agent, usually in the high temperature zones;

2) the indirect reduction index appears to be improved;

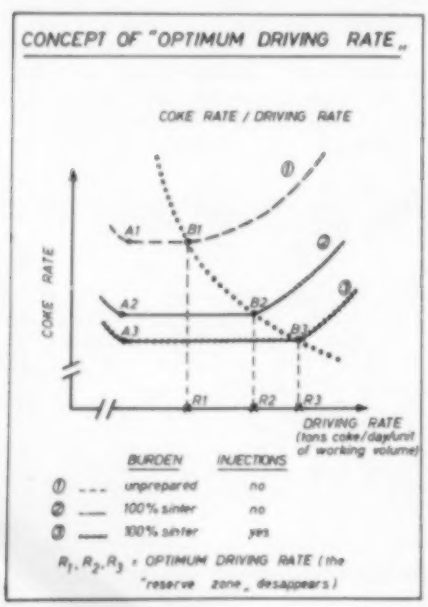
3) coke rate and production rate are drastically improved: the production rate went as high as 90 short tons per day per 1000 cu ft of working volume, and the retention time of the solids in the furnace is now shorter than 3 hr and could be further reduced, especially if the sinter were charged hot;

4) hydrogen injection appears to eliminate the solution loss reaction in the low-shaft furnace; and

5) hydrogen injection can compete with high top pressures up to a certain production level, thereby increasing the optimum driving rate as shown in Figs. 10 and 11.

There are no technological difficulties with injections through the tuyeres, particularly with gas, but fuel oil should be very well atomized so as to burn with a very short flame.

At Liège, investigations are being carried forward to improve injection techniques for large quantities of fuel oil and the injection of coke-oven gas and pulverized coals. In addition, a study of the qualities of cokes is to be undertaken.



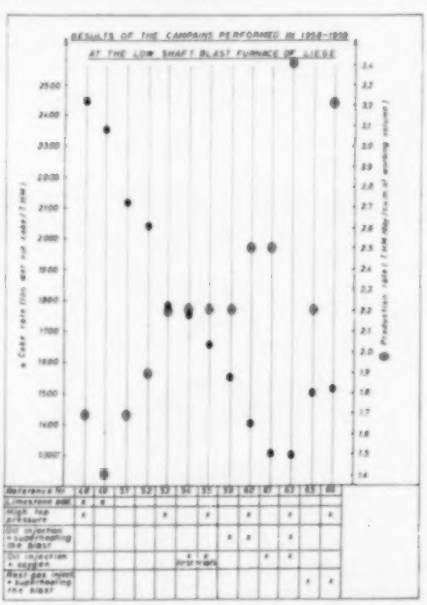


Fig. 10, Left, Concept of optimum driving rate. Fig. 11, Right, Results of low-shaft furnace campaigns.

THERMAL ASPECTS OF BLAST FURNACE FUEL INJECTION

Results obtained with pilot-plant blast furnaces must be extrapolated for application to commercial furnaces. Using the results obtained from the low-shaft blast furnace at Liège, Belgium, this paper presents some rules for correlating these results.

by A. M. Decker

Injections of solid, liquid, or gaseous fuel into the tuyeres of blast furnaces, studied intensively in several countries during the past several years, have given rise to further experiments with industrial furnaces or small pilot-plant blast furnaces[1-6]. These studies [6] have led to the conclusion that fuel injection could be economically interesting from the fuel consumption point-of-view, as well as from the productivity aspects of the furnace. But when trials are run in pilot plant furnaces, it becomes necessary to extrapolate the data to correlate with industrial blast furnace operation. The purpose of this paper is to formulate, with the data obtained from the International Low-Shaft Blast Furnace at Liege, rules with which pilot-plant results can be scaled up to commercial-size blast furnaces.

If the required coke rates were readily predictable, the correlation problem would be relatively simple. Unfortunately, the methods available at present are somewhat unsatisfactory. Statistical methods[7] introduce into the correlation formulas factors that influence the coke rate. In addition, it is necessary to obtain results which are not applicable to the fuel injection. And when methods based on heat balances are used,[8-12] it is necesary to make some assumptions as to the indirect reduction ratio, the action of hydrogen, the temperature of the top gas, and the heat losses, etc. Furthermore, the productivity of the furnace also enters as an additional unknown factor.

Calculation Method

In order to explain our method of calculation, consider the existence of three types of reactions:

$$r_{co}MO + r_{co}CO \rightleftharpoons r_{co}M + r_{co}CO_2$$
$$r_HMO + r_HH_2 \rightleftharpoons r_HM + r_HH_2O$$
$$RMO + RC \rightleftharpoons RM + RCO$$

The first two equations refer to the indirect reduction of the oxides by carbon monoxide and hydrogen and the third refers to direct reduction by solid carbon. In these equations, r_{co},r_H, and R represent the mean fractions of the metallic oxides reduced by carbon monoxide, hydrogen, and carbon respectively. It is possible to distinguish between these reductions, properly called direct reduction, and these indirect reductions followed by the solution loss, but this distinction is not necessary. Therefore, the two types of reduction can be grouped into the third reaction.

The analysis of top gas, especially its nitrogen content, and the consumption of carbon allows the calculation of the quantity of blast and the total quantity of oxygen coming from the iron oxides and the oxides of the other elements. The balance of hydrogen is then used to calculate a mean percent-

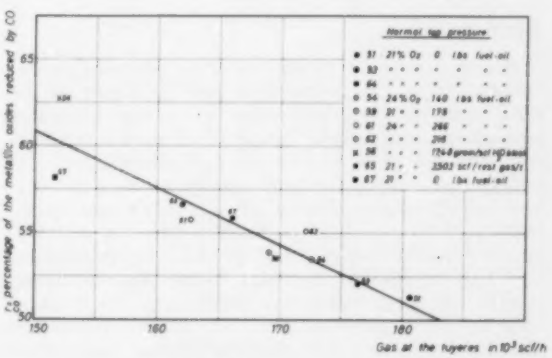
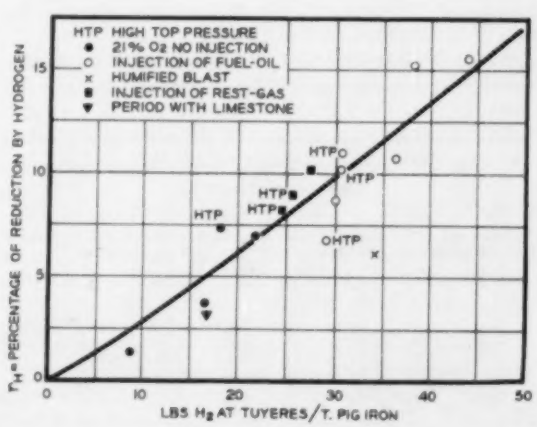


Fig. 1—Above, variation of r_{CO} with hourly gas flow at tuyeres. Fig. 2—Below, variation of r_H with wt H_2 in the system per ton of pig iron.



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age of the reduction by hydrogen, r_H. Knowing the oxygen content and the blast humidity, the carbon of the direct reduction can be calculated by subtracting from the total carbon consumed that carbon which carburizes the pig iron, that which goes into flue dust, and that which burns at the tuyeres. Included in the carbon which burns at the tuyeres is the amount necessary for the complete dissociation of blast humidity. The mean percentage of direct reduction, R, is inferred from the carbon of direct reduction. The mean percentage of indirect reduction by carbon monoxide can be found with the following equation, $r_{CO} = 100 - (R + r_H)$.

Objections could be raised that the mean percentages, as defined, are not meaningful. In fact, it is reasonable that Fe_2O_3 is totally reduced indirectly whereas MnO is reduced directly. However, these percentages provide the simplest method of characterizing a global state of reduction by the means of gas analyses, and since the calculation introduces no error from the thermal point of view, their use is justified.

The three reactions are endothermic; thus, they require heat that must be furnished by burning carbon at the tuyeres with the hot blast. In addition to the heat of reaction, carbon must also be consumed to melt the pig iron and slag, ensure the slag reactions, remove the CO_2 of the carbonates, remove the H_2O of the hydrates, dissociate H_2O of the blast or some of the hydrates, crack the hydrocarbons, and compensate the heat losses. Heat is also lost because the stack gas leaves the furnace with a certain sensible heat.

Application

If any two of the three reduction percentages are known, the third percentage can be found in the same manner that the carbon monoxide reduction percentage was found. Therefore, knowing the burden, the analysis of the iron produced, and the hourly heat losses, the fuel consumption of the furnace can be calculated in advance. All runs in the experimental low-shaft furnace utilized a sinter of rather constant composition[13]. Thus, with the burden and the method of charging being approximately constant, r_{CO}, r_H, and R will depend on the working rate and the different fuel injections, if constant thermal losses and iron composition are maintained.

Fig. 1 illustrates the variation of r_{CO} in relation to the hourly gas flow at the tuyeres without top pressure. Hourly gas flow[3,6,10,14] is taken as: [(volume of blast + volume of O_2 blast + 2 x volume H_2O + volume of H_2 fuel-oil) per ton of pig iron] x the hourly production of the furnace.

As can be seen in Fig. 1, the grouping of the points around the traced line is excellent, even though the

Table I. Predicted and Observed Results Obtained with Light Fuel-Oil Injection

Coke Consumption, lpd		Pig Iron Production, tph		Carbon Consumption from Coke & Fuel, Lb/THM			
Predicted	Observed	Predicted	Observed	Predicted	Observed	Δ, lb	Δ, pct
44.6	45.7	1.67	1.81	3544	3492	−52	−1.53
44.0	45.6	1.73	1.69	3368	3342	−26	−2.11
39.7	43.5	1.74	2.05	3010	2786	−224	−8.08
41.0	41.0	1.91	2.05	3108	3100	−8	−0.28
37.7	39.7	1.82	2.05	2994	2892	−102	3.80
40.0	41.7	1.56	1.54	3394	3482	+88	2.90

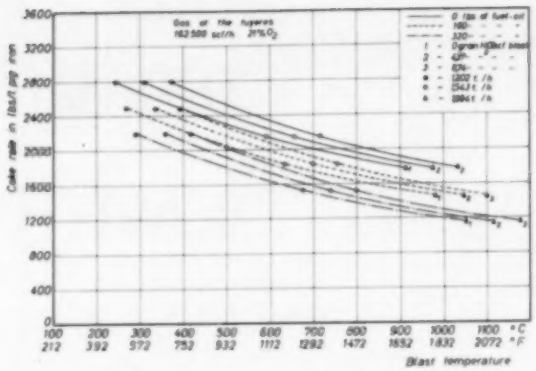


Fig. 3—Relation between coke rate and blast temperature for various levels of fuel-oil injection.

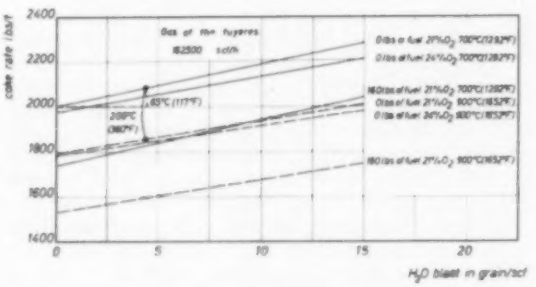


Fig. 4—Coke rate vs humidity in blast.

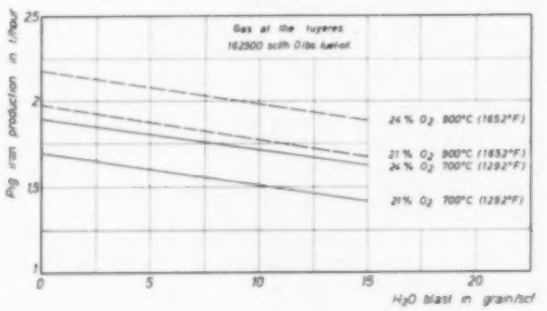

Gas at the tuyeres
162500 scfh 0 lbs fuel-oil
Pig iron production in t/hour
24 % O2 900°C (1652°F)
21 % O2 900°C (1652°F)
24 % O2 700°C (1292°F)
21% O2 700°C (1292°F)
H2O blast in grain/scf

Fig. 5—Output vs humidity in blast.

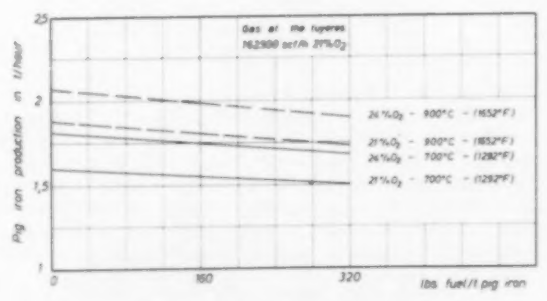

Gas at the tuyeres
162500 scf/h 21%O2
Pig iron production in t/hour
24 %O2 - 900°C - (1652°F)
21%O2 - 900°C - (1652°F)
24%O2 - 700°C - (1292°F)
21%O2 - 700°C - (1292°F)
lbs fuel/t pig iron

Fig. 6—Output vs fuel-oil injected.

points correspond to different runs with or without injection of fuel-oil, with injection of rest-gas, or with injection of blast moisture. For a variation of 10 pct, the ratio r_{co} varies within 5 pct which is relatively important. For a given rate at the tuyeres, without top pressure, it appears that r_{co} does not depend on the first approximation or the quantity of injected fuel-oil. Hydrogen does not seem to influence the indirect reduction by the carbon monoxide in the first approximation. Thus, r_{co} is fixed for a given gas flow. With Minette ore, r_{co} is about 55 pct for the nominal working rate; however, with a 100 pct self-fluxing sinter, this value increases to approx 60 pct. There is no correlation when r_{co} is expressed in relation to the carbon rate ($C_{coke} + C_{fuel}$) per hr, and it was also found that, with high top-pressure, indirect reduction by carbon monoxide varied slightly for a 23-pct change in flow rate while allowing a substantial increase in production without requiring an increase in coke rate.

Experiments have ascertained that the reduction by hydrogen, r_H, is nearly linear function of the weight of the hydrogen in the system per ton of pig iron. The points in Fig. 2 are relatively consistent, even though an inexact knowledge of the hydrogen content of the coke could cause variations in the hydrogen balance Top pressure apparently does not effect r_H, which would indicate that an equilibrium is established rapidly with a hydrogen content corresponding to a given temperature. With knowledge of the amount of oxides to be reduced, the fraction of hydrogen necessary for reduction can readily be calculated. In the experimental range employed, this was found to be approx 37 pct. At this point having values for r_{co} and r_H, R can be found by subtraction.

If the thermal losses of the furnace are known, the problem could be completely solved. During the early stages of these experiments, the mean value of the losses of the Liege Low-Shaft Furnace, were found to be approx 3.7×10^6 Btu per hr. After the furnace was relined, the losses were calculated to be 3.3×10^6 Btu per hr. These values are consistent with commercial blast furnaces when a minimum of 2 tons of pig iron are produced per hr. From the foregoing data it is possible to predict the required coke rate, assuming that:

1) r_{co} remains constant at a given gas rate,
2) the hydrogen content of the system remains constant,
3) the heat losses remain constant, and
4) a relationship exists between the temperature of the top gas and the blast rate at the tuyeres.

Predictions of the coke rate for various fuels were made with a IBM 650 computer. The following were considered constant in the preliminary runs without fuel: a mean burden, the oxygen content of the blast, the blast temperature, and the blast rate in the tuyeres.

Light fuel-oil

In addition to the previous factors, fuel-oil injection necessitates that either the quantity of the fuel-oil per ton of pig iron or the coke consumption per day must remain constant. The light fuel-oil used had a composition of 85 pct C and 13 pct H_2. For runs with top pressure, a uniform gas rate of 160,000 cu ft per hr was maintained.

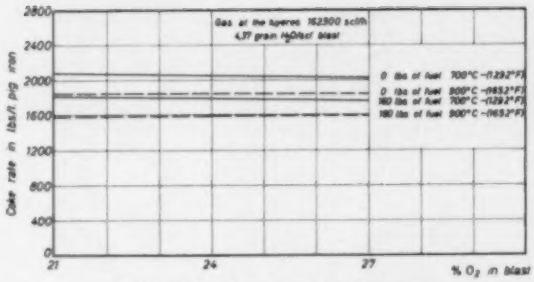


Fig. 7—Coke rate vs oxygen enrichment.

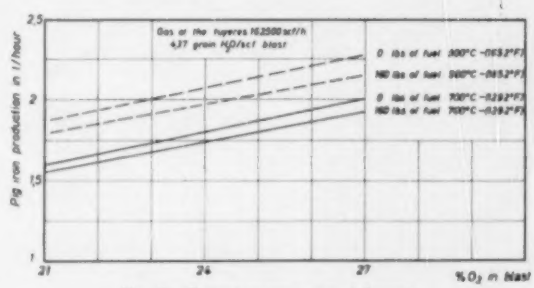
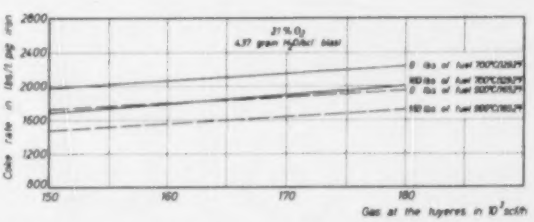


Fig. 8—Output vs oxygen enrichment.



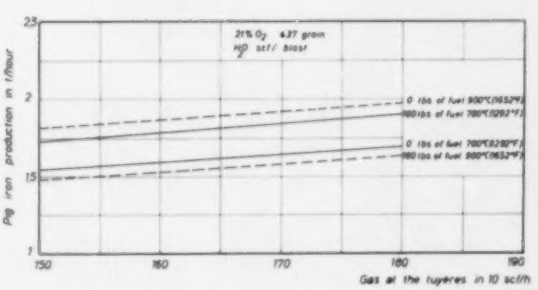


Fig. 9—Above, Coke rate vs quantity of blast at tuyeres. Fig. 10—Below, Output vs blast volume.

Table I illustrates the predictions made and the results obtained for coke consumption, pig iron production, and the coke rate per ton of hot metal (THM). In addition, the volume of both the blast and the gas, and the analysis of the top gas was calculated.

A certain amount of fuel can be injected without necessitating higher blast temperatures. This temperature increase is useful, but it is necessary only when it is impossible to use a lower combustion temperature. Fig. 3 shows the effect of the blast temperature on the coke rate.

Fig. 4 gives the variation of the coke rate with the humidity of the blast for two injection levels. For a moisture content of 4.37 grains per cu ft, the proportionality of coke rate and blast humidity indi-

cates that, in order to maintain a constant coke rate, it is necessary to increase the blast temperature by 117°F or, the equivalent of 6.5°C per grain of humidity in the blast. This temperature increase is necessary for the complete dissociation of H_2O at the tuyeres and yields a hydrogen content of 37 pct. According to Grieve,[15] Semik[19] reported hydrogen yields between 25 and 50 pct, and Grieve found yields of 40 pct in the blast furnace. Fig. 5 illustrates the variation of the hourly production of pig iron in relation to the blast humidity, for a constant bosh-gas rate.

Effects of fuel-oil rate on production are illustrated in Fig. 6. The influence of oxygen enrichment of the blast is illustrated in Fig. 7 and 8. Enrichment gives an inperceptible gain in the coke rate while allowing a considerable increase in production.

As illustrated in Figs. 9 and 10, a 10 pct increase in the gas rate at the tuyeres provokes an increase in the coke rate on the order of 7 to 8 pct.

Rest-Gas and methane

Injection of rest-gas, with the following approx composition, 59.7 pct CH_4, 14.7 pct N_2, 5.3 pct H_2, 13.3 pct CO, and 2.7 pct O_2, led to the same type of results as with light fuel oil—Fig. 11.

Concluding that methane acts in a similar manner as rest-gas and fuel-oil, Fig. 12 was obtained.

Table II. Influence of Coal Injection on Coke Consumption*

Temp, °F	Coal Injected, lb/THM	Coke Consumption, lb/THM[b] 21 pct O_2	Coke Rate Increase, lb/THM
1292	0	2000	188
	200	1812	
1652	0	1768	194
	200	1574	

* Same as Table II.

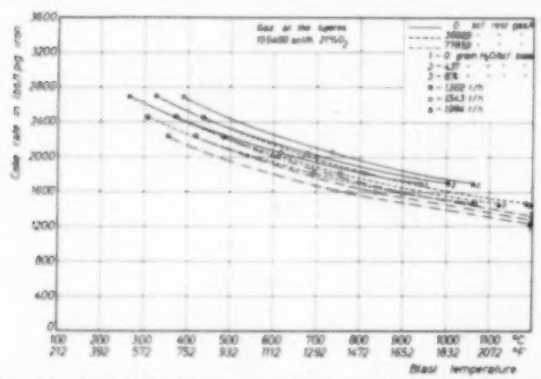
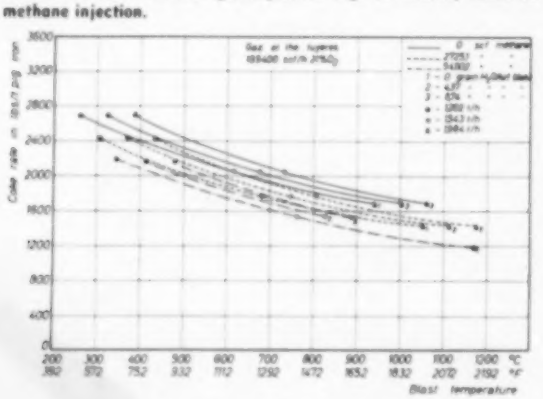


Fig. 11—Above, Relation between coke rate and blast temperature for various levels of rest gas injection. Fig. 12—Below, same for methane injection.



Pulverized coal

Complications arose when injecting coal, and the effects of ashes and eventual humidity must be considered. Table II presents the coke rate increase due to the injection of 200 lb coal per THM. At 3 pct humidity the injected coal containing 82 pct C, 3.4 pct H_2, 0.8 pct O_2, 1.2 pct N_2, 10 pct ash, replaces approx 166 lb of coke. It would be interesting to verify these results in the blast furnace. According to Loginov et. al.[5], injection of a pulverized coal representing 5 to 6 pct of the total coke consumption, decreases the overall consumption approximately the same percentage.

Industrial blast furnace

In the case of a blast furnace already attaining high indirect carbon monoxide reduction ratios of 65 to 70 pct, savings due to hydrogen reduction tends to be less than the values presented for the low-shaft furnaces. Direct reduction always has a certain minimum value; however, it is impossible to be precise on this matter without carrying out experiments, and for that reason, injections in an industrial blast furnace are currently being observed by the Centre National de Recherches Metallurgiques and the Cockerill-Ougree firm in Belgium.

Conclusions

Several trial runs in the low-shaft furnace with or without fuel oil and/or rest-gas injection, and with or without top pressure, have established relationships that define the indirect reduction percentages of carbon monoxide and hydrogen; these are:

1) r_{CO} depends principally on the gas rate at the tuyeres, and

2) the fraction of the reacting hydrogen remains constant at approx 37 pct.

Utilizing these two relationships, the coke rate and the hourly production can be predetermined for the low-shaft furnace. The same method is applicable for commercial blast furnaces and for various burdens if the variations of the r_H and r_{CO} factors for the particular furnace are known.

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FERROMANGANESE PRODUCTION WITH OXYGEN ENRICHMENT

Tests with oxygen enrichment of the blast of ferromanganese furnaces to 26.5 pct have shown a production increase of 29 pct and a reduction in the coke rate of 11 pct. Indications are that at higher levels of oxygen enrichment, additional improvements are possible.

by J. M. Stapleton, R. J. Flanagan,

R. L. Stephenson, and D. A. Fletcher

Oxygen in the blast for ferromanganese production has been used since May of 1957 at the Duquesne steel works of the U.S. Steel Corp. Considerable success in terms of increased production rates and decreased coke and stone requirements has been achieved with oxygen enrichment. The first year's commercial operations with the blast enriched to 26.5 pct O_2 has shown an increase in production of 29 pct along with a reduction in coke rate of 11 pct. Since June of 1958, oxygen enrichment has been increased in one blast furnace to above 30 pct in the blast. These trials lasted only a few months; however, they indicated that, at higher levels of oxygen enrichment, additional improvements in production rate and in coke savings could be obtained. From all indications, the level of optimum oxygen enrichment is well above 30 pct.

Although this is the first commercially-successful application of oxygen enrichment to the production of ferromanganese, the idea is not new. In a discussion of the future of oxygen enrichment of air for metallurgical processes at the 17th general meeting of the American Iron and Steel Institute in 1920, C. A. Meissner, then Chairman of the U.S. Steel Corp. Blast Furnace Committee, pointed out that, although there was some question about the value of oxygen in basic iron production, there should be definite gains from the use of oxygen enrichment for the production of ferromanganese.[1] Since then, oxygen enrichment has been tried successfully in Germany[2] and in the USSR[3, 4], but never have the benefits been realized to the fullest extent.

At Duquesne works of the U.S. Steel Corp. two blast furnaces are normally operated to produce ferromanganese. The lines and important dimensions of these furnaces are shown in Fig. 1. Both No. 2 and 3 furnaces have used oxygen enriched blast. The pertinent dimensions of No. 2 furnace are a hearth diam of 20.5 ft with a working volume of 28,919 cu ft (working volume to hearth area ratio = 87.6). The furnace has four stoves with 3 in. checker flues for a total heating surface of 485,412 sq ft.

Fig. 1—Ferromanganese furnaces at Duquesne works.

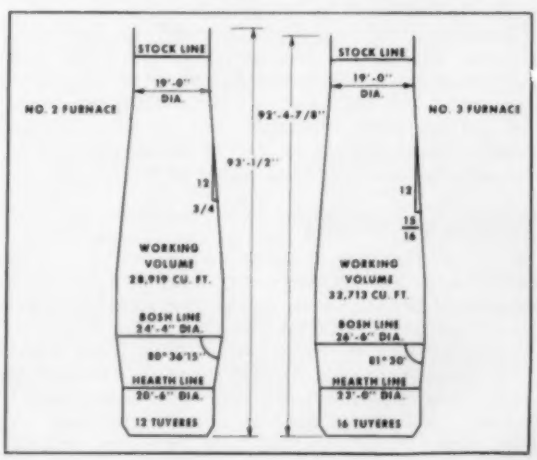

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Table I. Mn Ores Used in No. 2 and No. 3 Furnaces, Pct

Ore	No. 2 & No. 3 Furnace, 1957	No. 2 Furnace, 1958		
		Aug	Sept	Oct
Brazilian Merid	22.36	6.83	—	—
South African 44 (pct)	13.8	20.12	17.79	18.93
Oriental	7.97	37.64	28.16	23.75
Belgian Congo	8.06	21.44	25.10	2.24
Indian 40-44 (pct)	7.57	—	—	—
Indian 44-48 (pct)	8.94	13.97	15.93	29.03
Indian Over 48 (pct)	3.15	—	—	—
Rhodesian	1.38	—	3.83	1.04
Dongri	1.96	—	9.19	8.33
Brazilian Amapa	—	—	—	16.68
All Other	24.83	—	—	—

Table II. Analyses of Mn Ores, Natural Basis, Pct*

Ore	Moisture	Fe	P	Mn	Total Met.	SiO_2	Al_2O_3	MgO	CaO
Brazilian Merid	7.70	4.07	0.097	40.65	44.81	6.99	8.03	0.29	0.15
Brazilian Amapa	4.43	3.69	0.085	48.89	52.67	2.47	4.48	0.05	0.11
Oriental	1.03	6.51	0.144	48.80	55.45	9.01	1.59	0.63	0.83
Dongri	1.97	5.98	0.254	50.09	56.32	3.96	1.70	0.14	0.23
South African 44 (pct)	0.74	14.13	0.046	43.99	58.17	3.86	3.58	0.48	0.92
Indian 40-44 (pct)	4.29	10.56	0.084	40.81	51.45	5.23	4.46	0.23	0.18
Belgian Congo	3.62	1.87	0.113	49.78	51.76	3.95	4.11	0.09	0.19
Indian 44-48 (pct)	2.75	7.82	0.120	44.95	52.89	8.54	3.50	0.36	0.77
Indian Over 48 (pct)	2.44	4.87	0.191	48.74	53.80	6.18	2.15	0.23	0.25
Rhodesian	2.12	2.21	0.028	54.98	57.22	3.06	1.46	0.25	0.29

* Based on Average Analyses, Oct. 1958.

Table III. Typical Size Consists of Various Mn Ores, Avg Screen Analyses, Cumulative Pct

Ore	+ ½ In.	+ 3 Mesh	+ 10 Mesh
So. African 44 (pct)	83.7	88.8	94.4
Indian 40-44 (pct)	61.2	75.6	91.2
Belgian Congo	84.9	89.5	94.5
Oriental	56.4	71.5	89.6
Indian 44-48 (pct)	66.9	82.2	92.9
Brazilian Amapa	55.1	67.3	87.1
Dongri	38.2	64.6	90.6
Brazilian Merid	47.6	66.1	89.8
Rhodesian	78.3	84.3	90.9

No. 3 furnace has a hearth diameter of 23 ft with a working volume of 32,713 cu ft (working volume to hearth area ratio = 78.7). This furnace has four stoves with 2 in. checker flues for a total heating surface of 502,744 sq ft.

Raw materials

Ores used in producing ferromanganese vary widely in source, as well as chemistry and physical characteristics. It is, therefore, necessary to mix them in suitable proportions to obtain the proper chemical composition of the hot metal. The limited availability of the individual ores makes it necessary to change the burden mix frequently. Table I shows the ore mixes used on No. 2 and No. 3 furnaces during the various trial periods.

Variations in silica content between different ores causes variations in slag volumes and, in turn, production and coke rates. Table II shows typical analyses of manganese ores, while Table III presents the size analyses of various ores. Because of the coarseness of the ores, as well as the large volume of coke in the charge, movement of the burden is ordinarily quite smooth.

The coke used has approximately the following analysis: 87.0 pct C, 1.24 pct S, 9.80 pct ash, and 2.50 pct moisture. And the ash analysis is as follows: 48.47 pct SiO_2, 5.20 pct CaO, 27.04 pct Al_2O_3, and 1.33 pct MgO. Physically the coke is not particularly good, having an ASTM stability factor of only 38 pct and a hardness factor of 63 pct.

Furnace operations

Prior to the use of oxygen enrichment, No. 2 furnace normally produced about 300 tons of ferromanganese per day with a coke rate of 3760 lb per ton of hot metal (THM). No. 3 furnace produced 390 tons per day at a coke rate of 3430 lb per THM.

The ferromanganese is cast directly into cars at the furnace. These cars are approx 40 ft long and 8 ft wide with an all steel frame. The bottoms are lined with 4 in. of clay, one 2-½ in. course of ladle brick laid flat, 1 in. of clay, and a 4.5 in. service course of ladle brick laid on edge. The sides and ends of the car are lined with 4 in. cast iron plates (20 plates in total) and the joints are packed with clay. Each car will hold about 40 tons of ferromanganese filled to a depth of 8 to 9 in., with three cars required for the average size cast.

After filling, the cast cars are allowed to cool for 24 to 36 hr before being unloaded. Unloading is performed by a machine located over railroad tracks, followed by a primary crusher. From the primary crusher the ferromanganese goes via conveyors to screening stations.

Oxygen plant

Following a preliminary trial of three months duration to demonstrate the feasibility of oxygen enrichment, using oxygen supplied in tank cars, the U.S. Steel Corp. had installed at the Duquesne works an oxygen plant capable of producing 430 tpd of low purity oxygen (95 pct pure) and 70 tons per day of high purity oxygen (99.5 pct pure). This plant supplies oxygen to the intake of the turboblowers at a maximum rate of about 430,000 cu ft per hr at a delivery pressure of 2 to 2.5 psig. This is sufficient to enrich the blast of No. 2 and No. 3 furnaces to about 26.8 pct by volume. To insure a continuous supply of oxygen for at least 15 hr in case of a shut-down of the oxygen plant, about 6,500,000 cu ft is stored in liquid form in a reserve tank.

Oxygen is introduced into the blast after the venturi control meter at the inlet side of the turboblowers. Hence, the wind leaving the turbo-blower is at the desired enrichment. Automatic regulator controls keep the flow of oxygen into the turboblower intake at the desired proportion to the flow of air from the venturi. The total blast air is metered again after the blower, so that, by comparing the flow of air and oxygen to the total flow after the blower, the oxygen content of the blast can be calculated. Spot chemical analyses of the cold blast have shown that the per cent enrichment calculated from the two flow meters is accurate.

Operations with oxygen enrichment

As the oxygen content of the blast is increased, the burden must be increased commensurate with the demands of the furnace. The wind is reduced to keep the furnace moving properly, and the casting schedule is changed to accommodate the additional production of the furnace. At 26.5 pct O_2 in the blast, the time between casts was reduced from 6 hr to 5 hr and 20 min. At higher levels of oxygen enrichment, this was further reduced to 4 hr and 50 min.

Operating data for the two periods (Tables IV and V) show that, with oxygen enrichment, the production rate for No. 2 furnace increased from 300 to 398 tpd and that for No. 3 furnace, from 386 to 484 tpd. The coke requirement for No. 2 furnace decreased from 3765 to 3292 lb per ton of ferromanganese and that for No. 3 from 3430 to 3104 lb per ton of ferromanganese. An increase in hot-blast temperature (1505° to 1536°F) for the No. 2 furnace during the oxygen-enrichment period caused the reduction in coke rate for the No. 2 furnace (12.6 pct) to be greater than that for the No. 3 furnace (9.5 pct). This difference and the difference in wind rates for the two furnaces account for the greater increase in production rate for No. 2 furnace (32.7 pct) compared to that for No. 3 (25.7 pct).

In addition to the improvement in production rate and the decrease in coke and stone rates, other effects were noticeable with oxygen enrichment. The fuel value of the top gas increased from 121 Btu per cu ft to about 131 Btu (see Table VI). Slag volume decreased, with little change in the manganese content of the slag; consequently, the per cent of manganese recovered increased slightly.

The number of tuyeres changed per month increased from 7 per month during the normal air period to 17 per month during the 26.5 pct O_2 period.

Theoretical consideration

Experience has shown that oxygen enrichment of the blast is of much greater benefit in the production of ferromanganese than in the production of basic iron. The reason for this can best be demonstrated by comparing the operation of a ferromanganese blast furnace with that of a basic iron furnace. In a basic furnace, the gases in the shaft portion of the furnace reduce almost all of the higher oxides of iron (Fe_2O_3 and Fe_3O_4) to ferrous oxide (FeO) as well as reducing a large amount of this ferrous oxide to metallic iron (Fe). This type of reduction is termed *indirect reduction* and is usu-

Table IV. Operating Statistics, No. 2 & No. 3 Furnaces at Normal Air and 26.5 Pct O_2, 10 Month Avgs.

	No. 2 Furnace			No. 3 Furnace		
	Normal Air	O_2 Enrichment	Pct Change	Normal Air	O_2 Enrichment	Pct Change
No. Days in Period	303	306	—	303	306	—
O_2 of Blast, Pct	20.9	26.5	—	20.9	26.5	—
O_2 Entering Fce, cfm	9000	11000	+22.2	10900	12700	+16.5
Production, tpd	300	398	+32.7	386	484	+25.4
Coke Rate Lb/THM	3765	3292	−12.6	3430	3104	−9.5
Hot Blast Temp, °F	1505	1536	+2.1	1537	1540	+0.2
Wind Rate at Turbo-Blower, cfm	42900	41500	−3.3	52300	48000	−8.2
Hot Blast Pressure, psig	17.9	17.9	—	16.8	16.6	−1.2
Slag Vol. Lb/THM	1879	1629	−13.3	1758	1585	−9.8
Stone Rate Lb/THM	1485	1313	−11.6	1425	1204	−15.5

Table V. Hot Metal and Slag Analyses, No. 2 and No. 3 Furnaces, Normal Air and 26.5 Pct O_2

	No. 2 Furnace		No. 3 Furnace	
	Normal Air	O_2 Enrichment	Normal Air	O_2 Enrichment
O_2 Content of Blast, pct	20.9	26.5	20.9	26.5
Hot Metal Analysis, pct				
Mn	77.64	76.96	76.78	76.89
Si	0.58	0.68	0.65	0.67
P	0.244	0.238	0.235	0.225
S	0.033	0.031	0.031	0.030
Fe	15.57	15.17	15.44	15.33
Slag Analysis, pct				
SiO_2	23.12	22.93	23.61	23.24
Al_2O_3	20.81	21.11	21.41	21.24
CaO	32.47	35.24	34.95	35.01
MgO	10.85	8.97	9.66	9.09
Mn	7.96	7.45	6.68	7.36
S	2.39	2.32	2.42	2.36
Avg Slag Basicity				
Avg Base (CaO + MgO), pct	43.32	44.21	44.61	44.10
Avg Acid (SiO_2 + Al_2O_3), pct	43.93	44.04	45.02	44.48
Base to Silica Ratio	1.87	1.93	1.89	1.90
Base to Acid Ratio	0.99	1.00	0.99	0.99

Table VI. Top Gas Analyses and Fuel Value, No. 2 Furnace

Periods	O_2 in Blast, Pct	Analysis, Pct				Btu/Cu Ft of Top Gas
		CO_2	CO	H_2	N_2	
Normal Air	20.9	2.60	34.40	2.90	60.10	121
O_2 Enrichment	26.5	4.83	37.73	3.32	54.06	133

Table VII. Ferromanganese Heat Balance, No. 2 & No. 3 Furnaces, Normal Air and 26.5 Pct O_2

	No. 2 Furnace		No. 3 Furnace	
	Normal Air	O_2 Enrichment	Normal Air	O_2 Enrichment
1. O_2 content of blast, pct.	20.9	26.5	20.9	26.5
2. Total heat, Btu, per THM (sensible heat of blast plus heat of combustion of coke).	18,036,500	15,646,500	17,224,100	14,923,800
3. Heat consumed in hearth, Btu per THM (for reduction of MnO and heating slag and metal above 2000°F).	5,651,100	5,333,600	5,448,100	5,311,300
4. Heat consumed in hearth, pct of total.	31.3	34.1	31.7	35.6
5. Heat consumed in stack, Btu per THM (heating charge to 2000°F).	4,989,200	4,467,000	4,690,100	4,264,000
6. Heat consumed in stack, pct of total.	26.7	28.5	27.2	28.6
7. Heat lost, Btu per THM (sensible heat of top gas, cooling and radiation).	7,396,200	5,845,900	7,085,900	5,348,500
8. Heat lost, pct of total.	41.0	37.4	41.2	35.8

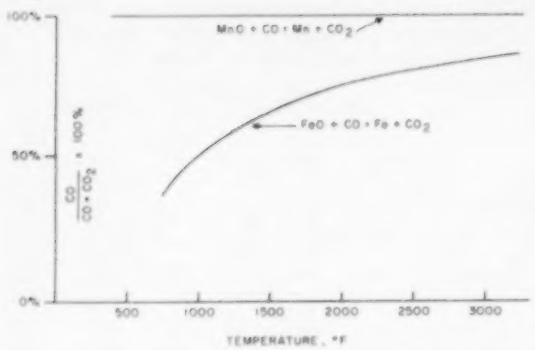

Fig. 2.—Equilibrium values of CO:CO + CO_2 for indirect reduction of FeO and MnO.

ally expressed by the following chemical equation:

$$CO + FeO \longrightarrow Fe + CO_2.$$

This reaction is exothermic, producing about 122 Btu for every pound of iron. Consequently, the larger the percent of iron that is reduced in this manner, the lower the total heat requirement.

As the materials in the furnace increase in temperature, however, the amount of CO (as percent of CO + CO_2) required for this reaction increases so that this reaction (indirect reduction) stops. Fig. 2 shows the amount of CO (as pct of CO + CO_2) required at various temperatures.

To continue reduction, either more carbon must be gassified by carbon dioxide or carbon must react directly with iron oxide in the hearth. In both instances the overall thermal and chemical results are the same. Such reduction is called *direct reduction* and is expressed by the following chemical equation:

$$C + FeO \longrightarrow Fe + CO.$$

This reaction is endothermic, requiring about 1130 Btu per pound of iron. Consequently, the larger the amount of iron that must be reduced in this way, the higher the heat requirement becomes. Normally in a basic blast furnace, less than half of the iron is reduced in this manner; so, the heat requirement is not excessively large. In contrast to this, very little indirect reduction is done in the stack of a ferromanganese furnace, and almost all of the reduction is done in the hearth.

Although the higher oxides of manganese (MnO_2 and Mn_2O_3) are reduced to manganous oxide (MnO) by the furnace gases, manganous oxide cannot be reduced to manganese in this way. Fig. 2 shows that the percent of CO in the gases would have to be greater than 99.9 pct after reduction of manganese, for all temperatures existing in the blast furnace. Since indirect reduction is impossible, all of the manganous oxide must be reduced by direct reduction. This is expressed by the following chemical equation:

$$C + MnO \longrightarrow Mn + CO.$$

This reaction is even more endothermic than that for ferrous oxide, requiring about 2130 Btu per pound of manganese. Consequently, in a ferromanganese furnace the heat requirements in the hearth are far in excess of that required in a basic iron furnace.

Another factor which augments the differences between these two processes is the difference in the temperature of direct reduction of iron oxide and manganese oxide. Although iron is almost completely reduced (over 99 pct) at 2000°F, the reduction of manganese is not nearly complete until the reaction temperature exceeds 2750°F. Thus, for every pound of manganese, not only is more heat required—2130 Btu as compared to less than 1130 Btu—but this heat must be available at a higher temperature—2750°F as compared to only 2000°F. Because the temperature of the furnace gases must be well above the temperature of the solids heated by these gases, the major portion of the ferromanganese furnace heat requirements are at a temperature above 3000°F.

To produce sufficient heat at this high temperature, large amounts of coke are required, even though a high percentage of the heat below 3000°F must be wasted. By the use of even higher hot blast temperatures than now used, more heat can be made available above 3000°F; however, by the use of a blast enriched with oxygen, not only can a greater amount of the heat be made available above 3000°F, but less of the total heat will be wasted. For example, an increase in the hot blast temperature from 1500° to 1800°F, would increase the total heat output for every pound of carbon burned at the tuyeres from 7200 Btu to 7700 Btu and would increase the amount of heat available above 3000°F from 1700 Btu to only 2150 Btu. On the other hand, an increase in the oxygen content of the blast from that of normal air (20.9 pct) to 26.5 pct would reduce (rather than increase) the total heat output for every pound of carbon burned at the tuyeres from 7200 Btu to 6800 Btu, but would increase the amount of heat available above 3000°F from 1760 Btu to 2200 Btu. Thus, by enriching the blast with oxygen, a much greater proportion of the total heat generated is available for the smelting operation and less of the heat is wasted.

Heat balances for the periods of normal operation and for the periods at 26.5 pct oxygen in the blast (Table VII) show that, with oxygen enrichment, the percent of heat used in the hearth increases and the percent lost decreases. For No. 2 blast furnace, enriching the blast to 26.5 pct oxygen lowered the heat loss from 41.0 to 37.4 pct and for No. 3 blast furance lowered the heat loss from 41.2 to 35.8 pct. Because this loss is still well above that for a basic iron furnace (about 30 pct), it is quite probable that even higher levels of oxygen enrichment could be used before the gas leaving the hearth could no longer properly heat and prepare the material in the stack. Recent tests with oxygen-enrichment beyond the 26.5 level indicate that these predictions are correct.

Conclusions

On the bases of these trials, it appears that unless some unforseen circumstances arise, it should be feasible to increase the level of oxygen enrichment to as much as 38 to 40 pct before all of the benefits of oxygen enrichment are obtained.

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COKE-OVEN GAS INJECTION

Tests showed that coke-oven gas, containing hydrogen sulfide, could be injected successfully into the tuyeres of a merchant iron blast furnace. Increased metal production and decreased coke consumption resulted; hence, the practice of gas injection was adopted on a production basis.

by E. R. Dean and R. A. Powell

Early in 1958, the idea of using coke-oven gas as a fuel injected into the blast furnace was discussed among the operating, research, and engineering personnel of Pittsburgh Coke & Chemical Co. Several ideas for the introduction of the gas were set forth, including: introduction into the hot-blast main, injection into the bustle pipe, injection into the hot-blast stoves, or introduction of the gas directly into the furnace bosh above the tuyeres.

In 1959, discussion with Koppers Co. began, and definite plans were made to inject coke-oven gas, containing small amounts of hydrogen sulfide, into the blast furnace through the tuyeres. Calculations by O. R. Rice of Koppers Co. and J. S. Mackay of Pittsburgh Coke & Chemical Co., covering the chemical reactions and heat balances were used as a basis for all discussions. Several conferences between the two firms developed features of a control system suitable for the coke-oven gas tests.

The blast furnace into which the coke-oven gas was to be injected has a 22-ft diam hearth with 29,307 cu ft of working volume and 12 tuyeres; it is equipped with three 22x100-ft hot blast stoves with a total heating surface of 231,000 sq ft.

The compressor station is located 1100 ft from the blast furnace. At the end of an existing supply line, a tank was installed as a pulsation dampener to smooth out the flow from the reciprocating compressors and provide a convenient point to drain off condensate. On the discharge side of the tank, an 8-in. supply line, provided with valves for convenient purging, was installed.

An air-operated flow control valve, operated in response to a manually-set recording controller, was located in the pyrometer room. For the experimental period, automatic proportioning of coke-oven gas to cold blast air was not provided, al-

Table I. Summary of Principal Data

	Base Period	Gas Test Period	Diff.	Pct Increase or Decrease
Daily prod, NT	888	972	+84	+9.5
Coke rate lb/NT (2.5 pct moisture)	1,718	1,506	−212	−12.4
CO gas, cfm	—	2,250		
CO gas in blast, pct		4.1		
Blast temp, °F	1,351	1,499	+148	+11.0
Moisture, grains/cu ft	10.4	9.8	−0.6	−5.8
Furnace delays, min/day	53	55	+2	+3.8
Top Gas				
CO/CO_2 ratio	2.09	1.97	−0.12	−5.7
Btu/cu ft	101	104	+3	+3.0
H_2, pct	3.4	5.5	+2.1	+62
Flue dust, lb/ton (dry)	138	75	−63	−45.7
Slag basicity	0.96	0.96	−0.04	−4.1

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Table II. Chemical Analyses, Pct

	Raw Materials						
	Karl Ore	Wilpen Ore	Swedish Pellets Ore	Kiruna Basic Ore	Dolomite	Flux-Stone	Coke
Fe	50.62	50.36	66.24	65.16	—	—	0.42
P	0.056	0.062	0.010	0.059	0.010	0.015	0.017
Mn	1.01	0.96	0.09	0.05	—	—	—
SiO_2	9.86	7.05	0.33	2.64	0.17	0.23	4.46
Al_2O_3	1.79	1.70	0.70	2.29	0.11	0.08	2.72
CaO	0.76	0.59	0.48	1.35	30.63	52.81	0.50
MgO	—	—	—	—	22.37	2.26	—
Burden, pct	35	35	20	10	—	—	—

	Flue Dust		Slag			
	Base Period	Gas Test Period	Base Period	Gas Test Period	Coke Oven Gas	
Fe	49.58	50.22	—	—	CO_2	1.9
SiO_2	10.82	9.97	34.8	36.0	Ill,	3.4
Al_2O_3	2.21	1.84	13.0	12.0	O_2	—
P	0.051	0.043	—	—	CO	7.3
Mn	0.68	0.55	—	—	H_2	56.3
CaO	4.82	5.69	42.5	46.0	CH_4	27.8
MgO	0.43	0.38	8.0	4.0	N_2	3.1
S	0.31	0.29	2.1	2.2	Btu cu ft	541

Table III. Physical Tests of Raw Materials

Ore	1 in.	½ in.	¼ in.	⅛ in.	20 M	Thru 20 Mesh
Karl	21.3	19.1	13.6	10.1	10.1	25.8
Wilpen	13.6	17.9	25.6	14.5	15.0	13.4
Swedish Pellet	75.0	19.3	4.7	1.0		
Kiruna Basic	12.1	12.9	20.0	18.0	31.7	5.3

Coke	Stability	Hardness	Ash, Pct	C, Pct	H_2O, Pct
Base Period	48.7	66.2	9.5	89.9	8.2
Gas Test Period	51.9	67.5	9.3	90.5	8.78

Fluxes	On 2 in.	On 1 in.	½ in.	¼ in.	Thru ¼ in.
Limestone	40.5	45.4	8.5	3.5	2.1
Dolomite	31.0	50.7	12.3	4.0	2.0

Table IV. Derivation of Coke Saving

	Base Period	Gas Period
Iron production, NT	4,400	4,858
Coke charged, incl. moisture, lb	8,101,200	7,825,600
Coke rate as recorded, lb/NT	1,825	1,611
Avg moisture in coke, pct	6.2	6.78
Coke rate (2.5 pct moisture), lb/NT	1,718	1,506
Coke saving (2.5 pct moisture), lb/NT	—	212
Avg gas rate, cfm	—	2,250
Estimated minutes gas off	—	344
Total gas, M cu ft*		15,426
Total coke saving (212 x 4,858), lb		1,029,896
Coke saving/M cu ft gas (1,029,896 ÷ 15,426), lb		67

* (5 × 1,440 − 344) 2,250 = 15,426,000 cu ft

though it would be essential for a production-scale installation. An air-operated safety shut-off valve, located adjacent to the flow control system, was arranged to stop the gas flow upon a signal from pressure-sensing devices.

Gas was supplied to six 3-in. downlegs by a 6-in. circle pipe, located at the walkway, above the bustle pipe. The pipe included plugged connections for six additional downlegs. As a precautionary measure, individual air-operated safety shut-off valves were located in each of the six downlegs. The need for such valves in any particular installation is determined by contaminants in the gas to be handled.

To balance the flow between the tuyeres, the downlegs included orifice plates for use with manometers, which were considered a useful tool during the entire test period. Modified face plates were designed for the tuyere stock elbows so as to locate both the lance gland and the peep sight on the horizontal centerline of the tuyere.

Each lance assembly consisted of a 1.5 in. type 347 stainless steel pipe with tee and valve to permit rodding-out, if required. A manual shut-off valve and flexible hose connected each lance to a downleg. The lance tip was set approx 8 in. back from the tuyere nose. Each lance was approximately centered in the tuyere by means of a *chair* made from a short section of the same pipe and welded to the lance.

No tuyeres were burned as a result of using gas; however, one lance tip was burned upon contact with slag.

The design incorporated a number of safety shut-off valves, each of which was controlled by a manual pushbutton station energizing a solenoid valve. After setting the flow controller to about half of the scheduled flow rate, the main and individual shut-off valves were opened in sequence and then the flow was brought up to full rate.

Procedure

Original efforts to hold operating variables constant were thwarted by ore stocking problems and the demands of the merchant iron business. During one month, gas flow rates were adjusted between 1000 and 3600 cfm, and the behavior of the furnace was observed. At the end of the month, it was believed that the furnace could be operated under closely-controlled conditions for a period of two weeks.

During the first week, a five-day period without gas injection was selected as the base period. During the second week, a five-day period with gas injection through six of the twelve tuyeres was used to compare with the base period. A summary of the principal data obtained is shown in Table I. Tables II and III illustrate the chemical and physical analyses of the raw materials, slag, and flue dust. Table IV shows a derivation of the coke savings.

Conditions set for the operation of the furnace for the comparative periods are as follows:

Base period: The B furnace was adjusted to a mold-iron burden with silicons in the 1.60 to 1.80 pct range. The blast volume was set and maintained at 56,000 cfm. Ore-to-coke ratio, blast temperature, and blast humidity changes were held to the minimum necessary for silicon control.

Test period: The furnace was operated the same as for the base period, except for the use of coke-oven gas at a flow rate of 2250 cfm—approx 4 pct of the blast volume. The blast temperature was set and held at the maximum available—1500°F. Ore burden and gas volume were held constant. Adjustments of the coke or changes in blast humidity permitted silicon control in the iron.

Adjustment for the use of gas at the 4 pct blast volume was made in the 12-hr period immediately following the base period, with reduction of the coke unit being made 6 hr before turning on the gas. Blast temperature was increased by 50°F, or the approximate equivalent decrease in blast moisture of 2 grains was made, with the addition of each 1 pct of gas to the blast volume. This amount was considered necessary to compensate for the chilling effect of the gas in the tuyere zone.

Observations

Although the comparative periods were short, substantial benefits were indicated by the use of coke-oven gas at 4 pct of the blast volume. The coke rate decreased from 1718 to 1506 lb—a saving of 12.4 pct or 212 lb. In addition, daily production increased from 888 to 972 tons of hot metal or a gain of 9.5 pct.

During the base period, there were occasions when the furnace resisted the use of blast temperatures in the 1400° to 1500°F range without the use of prohibitive moistures. This did not occur during the gas test period, tending to confirm that the coke-oven gas will permit the use of extremely high blast temperatures without the accompanying loss of coke due to moisture additions.

Present practice

Subsequent to the test period, the furnace has been equipped for injection through all tuyeres. Gas has been used as standard practice ever since, although natural gas has been substituted for coke-oven gas due to a plant shortage brought about by lower coke production.

Although the furnace has not been operated under controlled conditions, designed to determine coke savings, operating results are highly pleasing. Generally speaking, when producing the full range of merchant irons, including spiegeleisen, the combination of gas and higher blast temperatures have reduced coke requirements by 15 pct and increased the production potential by more than 10 pct.

PLASMA FOR EXTRACTIVE METALLURGY

Plasmas or ionized gases, existing at temperatures as high as 50,000°C, constitute the highest controlled temperatures known. At these temperatures, reactions occur about which we have little or no knowledge. This paper explores the various possibilities of using plasma devices for extractive metallurgy.

by Paul M. Tyler

Plasma or ionized gas is commonly called the fourth state of matter. Because most of the stars, including our own sun, are in the plasma state, the first scientists to be interested in plasma were the astrophysicists. During the last decade, however, physicists and engineers in other fields have been increasingly concerned with ultra-high temperatures.

The broad domain of plasma phenomena extends far beyond the frontiers of present human knowledge; hence, several millions of dollars a year are already earmarked for basic studies of the nature and behavior of plasma flames. The National Bureau of Standards' plasma research program alone costs almost $1 million a year and may soon be doubled.[1]

Competent investigators are actively at work exploring possible applications of plasma devices to the field of extractive metallurgy. Reports on this work will be awaited with interest, but they are not likely to be issued in the near future. The situation is aptly described in the following sentence from a letter received in reply to the writer's request for more information, "We are extremely enthusiastic about application of arc plasmas to extractive metallurgy, but, unfortunately, the work we are presently engaged in is very proprietary in nature."

Plasma generating devices

Plasmas are produced, especially in low density gases, with the aid of shock tubes, exploding wires, solenoidal magnetic fields, and various electronic or thermionic devices and techniques. However, at present the electric arc torch is the only practical means of generating and maintaining a large flow of gas heated to temperatures ranging much above those available from chemical flames. Several of these electric arc devices or plasma jets can be operated for indefinitely long periods to generate almost any desired temperature from below 4000° to 10,000°C. For brief periods, temperatures as high as 50,000°C have been measured.[2]

Plasma-arc torches are of two principal types. The stream of high-temperature gas or vapor from either of these looks very much like the flame from the more familiar plumber's blow torch or an oil burner. As distinguished from an ordinary low-intensity open arc, a plasma jet is formed when the current density, dissipated on the surface of an arc electrode, is greater than can be removed by radiation and conduction alone.

In the confined electric arc plasma jet, one form of which is shown in Fig. 1, a constricted arc is produced by confining all or part of the arc column in a chamber or nozzle into which a flow of relatively cold liquid or gas is introduced under pressure. The design and shape of the arc enclosure are vital factors governing successful operation. A constricted arc,thus, differs from conventional arcs, which are established in a relatively quiescent gas medium, and which may be operated either in the open atmosphere or in an enclosure without appreciable influence on their behavior and properties.

The high-intensity arc is a special type of arc discharge. It differs from the ordinary open arc in that the anodic current density is substantially higher. Most of the input energy is transferred directly to the anode face, instead of emerging from the conduction column. In its original form, which is a modification of the Beck arc employed in high-powered searchlights and motion-picture projectors, the material of the anode is rapidly vaporized and escapes from the arc as a stream of plasma or *tail flame*. Instead of using flame-supporting materials in the core of a carbon electrode, Sheer and Korman developed a consumable anode which would furnish feed material for desirable chemical reactions. In contrast with other

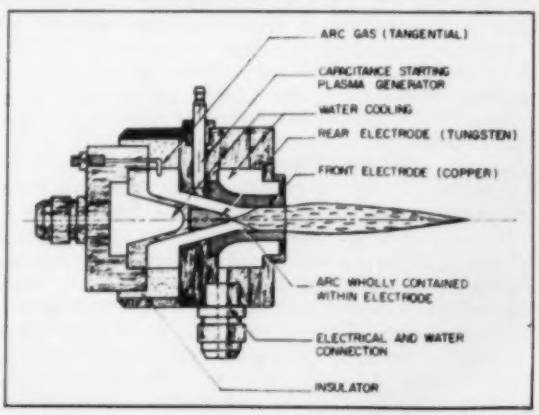

Fig. 1—Vortex stabilized plasma jet. Courtesy Plasmadyne Corp.

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consumable electrode processes, the high-intensity arc or *Hierarc* process is not limited to the treatment of electrically-conductive materials. Nonconductors, when suitably blended with a small percentage of carbon to provide electrical conductivity, may constitute 75 to 85 pct of the anode. Instead of manufacturing the feed material into a more or less conventional electrode, the high-intensity arc may be established from the surface of a mass of unconsolidated feed material in a graphite crucible. By replenishing the crucible charge at the same rate as it vaporizes, the process can be made continuous. A third modification, using the same energy-transfer phenomena, is the use of a porous anode. The material of the porous structure is not itself consumed, for the gas stream, which is needed in the anode sheath region in order to feed the arc, is supplied by the transpiration of fluid through the permeable anode.

General principles

Among the inherent characteristics of plasma devices and technology which may be of interest to extractive metallurgists are: 1) easy attainment of controlled ultra-high temperatures, 2) clean flames composed of a single element or of almost any desired mixture of elements, 3) high-velocity flow, 4) phenomenally-high heat transfer rates, 5) possible development of high pressure, high-temperature environments, 6) high concentration of energy in small space, and 7) possible elimination of containment problems.

Notwithstanding the rapidly growing interest in high temperatures as a new tool in the process industries, we still know very little about what happens or can be made to happen in the plasma domain. Synthetic chemists and research people have demonstrated that reactions do happen above 5000°K which are quite different from those observed at lower temperatures.[1]

Thermodynamic calculations indicate that electric current could be employed more efficiently in breaking chemical bonds by simple thermal action than by electrolysis in liquid or fused salt baths. In the new domain of much higher working temperatures, it is perfectly feasible almost instantly to convert any crude ore or even the most refractory mineral into a vapor composed of ionized atoms, completely free from chemical combination.

The objective of making a satisfactory recovery of one or more elements separately in a pure metallic state has not yet been achieved by condensation from a plasma flame. However, new methods of *freezing* chemical equilibrium compositions formed at high temperatures have been perfected to a notable degree. De Laval nozzles, for example, provide a continuous cooling rate of 10^6 °K per sec, and even higher discontinuous rates may be possible by other techniques. Such sophisticated quenching techniques could perhaps be employed for the fractional condensation of pure metal vapors.

Ultra-high temperatures obtainable with a plasma jet are of little or no value in extractive metallurgy unless back reactions can be prevented or adequately controlled. It has been said that "the science and technology of metal reduction is 10 to 20 years behind that in physical metallurgy, due to a lag in basic research".[2] This charge, however, does not imply that we cannot do any needed metal extraction job, only that we ought to find ways to do it better and more cheaply. Possible employments of plasma jets in extractive metallurgy are not dependent alone on the adoption of novel processes to make use of unfamiliar vapor-phase chemical reactions.

Another novel feature of plasma metallurgy is that composition as well as temperature-pressure conditions can be carefully controlled. There are no products of combustion, as in chemical flames, and contamination from furnace walls or other outside sources can be avoided. Inasmuch as the plasma is a conductor of electricity, it can be compressed and held tightly in a *magnetic bottle*. It can also be focused or otherwise manipulated by techniques based upon the new science of magnetohydrodynamics (MHD).

Plasma devices favor a high speed flow process rather than prolonged treatment in a large bath; thus, they not only eliminate the need for superrefractories for furnace construction, but they also make it feasible to operate with a small reaction zone and still maintain high capacity. Operations can be conducted in the open or in an unlined sheet-metal chamber whose walls are far enough away to be almost completely insulated from the hot flame by an intervening mass of relatively cool gas. Thus, radiation losses are minimized, and because the mass of material in process at any moment is quite small, the operation is flexible and readily controllable.

The enormous concentration of energy within a small space simplifies the design of equipment. In an aluminum reduction cell, for example, where the voltage is low and current density relatively high, the cathode absorbs no more than 5 kw per sq ft. By contrast, the anode diameters for a plasma jet may range from about 0.5 in. for a small 40-kw unit to 3 in. for a 3000 kw unit. The concentration of power per sq in. therefore, is 10,000 to 15,000 times as great.

High-Intensity arc process developments

Most of the pioneering research in plasma metallurgy has been conducted with high-intensity arcs by Charles Sheer and associates at the Vitro Laboratories div., Vitro Corp. of America, West Orange, N. J. According to Dr. Sheer (private communication) the following types of treatment have been demonstrated to be feasible:

1) Thermal decomposition,
2) Vapor-phase halogenation,
3) Vapor-phase carbothermic reduction, and
4) Liquid-phase carbothermic reduction.

Thermal Decomposition: In the original Sheer-Korman or Hierarc process for treating refractory raw materials, the ore was mixed with only sufficient carbon to provide electrical conductivity after being formed into a baked electrode. Later, the unconsolidated ore-carbon mixture could be fed into a graphite crucible and still serve as anode, thereby avoiding the expense of manufacturing a consolidated rod-type electrode.

The plasma flame from either form of anode consists predominantly of elemental vapors, including of course, a small percentage of carbon vapor—approx 15 to 20 pct. The product of the flame, when allowed to condense naturally in air, consists of a mixture of oxides with carbon monoxide gas passing off. While complex compounds such as euxenite

or refractory silicates might be present in the raw charge, it was found that back reactions were confined to the development of simple oxides, or even suboxides. The total residence time of free atoms and ions in the high-velocity flame is too short for polyatomic collisions. Rhodonite, for example, yields finely divided MnO and SiO_2 and beryl yields BeO, Al_2O_3, and SiO_2.

In the mass spectrograph it is possible to distinguish charged particles of differing mass. Various ways have been tried to segregate the oxides by electrostatic, magnetic, and fractional condensation methods. Some progress has been made in separating them according to their respective dew points by locating the bag filter for collecting the condensed solids at a point in the stream where the temperature is above the dew points of the undesired elements or compounds. However, it has thus far proved more practical merely to collect most of the oxides as a mixture and then to treat them by a second process.

In principle, the two-stage approach is the same as that of the melt-quench-leach method of attacking refractory ores. The only difference is that the reactions occur in the vapor phase, and that the intermediate product is an ultra-fine powder which is highly reactive. Experience has shown that the solubles dissolve very rapidly, and that the insolubles are easily filtered out. Although the particles are exceedingly fine—only 0.035 to 0.050μ in diam—they do not slime. Because of their highly disordered lattice structure, some of the fumed oxides may display anomalous solubilities in certain reagents. For example, the silica end fraction from the dissociation of zircon may be dissolved away from the zirconia by cold HCl.

Vapor phase halogenation: By injecting halogen gas into the plasma flame, it is possible to convert oxides to halides in the vapor state. The presence of carbon in the high-intensity arc undoubtedly helps this reaction. The boiling points of many chlorides are so widely separated that they can be condensed fractionally in a series of chambers maintained at suitable temperatures. The chlorides of Be, Fe, Al, Cb, Si, etc. may be caught at temperatures below 500°C. The technical feasibility of this approach has been demonstrated on such *ores* as beryl, kaolin, and borax. Even chlorides having almost the same boiling point, e.g. $CbCl_5$ and $TaCl_5$, can be separated by selective condensation because their different concentrations in the ore affect their relative dew points. This method may prove commercially suitable for treating euxenite and other complex or refractory minerals.

Chlorination processes have become more and more important industrially, owing to wider use of the Kroll process and fused-salt electrolysis. Although the plasma environment is not essential for the chlorinating reactions, it causes the reactions to take place many orders of magnitude faster than is possible in a fixed bed, and it also avoids the serious corrosion problems involved in the containment of hot halogen gas. Quantitative yields have been obtained consistently from complex silicates, as well as ordinary ores, treated by this new technique.

Vapor phase carbothermic reactors: Since carbon is the cheapest and most readily available and widely used reducing agent, it was a reasonable expectation that additions of carbon to the plasma flame might inhibit the reoxidation of metallic elements. Historically, the smelting of base-metal ores has been predominantly dependent upon the reduction of metal oxides with carbon at suitably high temperatures, preferably those high enough so that the undesired elements can be largely separated in the form of fluid slag. However, there are a number of interesting and useful metals whose oxides are not amenable to simple treatment by ordinary furnace methods. For the oxides of Al, Mg, Be, B, Si, Mn, Ti, Zr, and quite a few less-common metals, the minimum temperature required to promote the carbothermic reduction reaction is above the range of conventional pyrometallurgical equipment. Since plasma jets have no such temperature limitations, any of these metals can be liberated and, as the vapor stream cools down, the oxygen would tend to combine to form carbon monoxide.

The plasma flame generated in a high-intensity arc, fed by a consumable electrode composed of metal oxide and carbon in stoichiometric proportions, actually does consist of metal vapor and carbon monoxide. The absence of detectable amounts of metallic-oxide vapor in the flame has been confirmed spectroscopically in test runs for Al, Mg, and Be.

The principle of this process is almost identical with that of the Hansgirg process which was employed with meager success in California during World War II. Vitro succeeded in recovering 40 to 70 pct of the metal content in pure form simply by impinging the plasma flame on a water-cooled plate or by injecting cold hydrogen gas. These results are better than those reported for the Hansgirg process' but are still not good enough. Higher yields should be possible with more efficient shock-cooling, but until we know more about quenching kinetics, any prediction which has not been confirmed by actual experimentation must be viewed with caution. However, the relatively long and narrow high-velocity flame from a plasma device provides a more favorable configuration for rapid quenching, and the small mass of material in process at any moment of time would seem to eliminate the serious explosion hazards that plagued the Hansgirg operations.

Metal carbides and certain other compounds which are less likely to react with carbon monoxide than reactive metals have been produced successfully in plasma jets in several laboratories. The goal of pure metal recovery, although less easily attained, is still being sought. One proposal is to collect the metal produced by carbothermic reduction in a molten pool under conditions somewhat comparable to those in a vacuum melting furnace. Theoretically, if the flame were operated at reduced pressure and directed downward so as to impinge on the surface of the molten metal, the metallic vapor might be made to condense into droplets which could be instantly accepted by the surface of molten metal. The carbon monoxide would be deflected and eventually pumped out of the system. A variation of this proposal is to submerge the arc and tail flame in a molten salt bath or supernatant slag.

Liquid phase carbothermic reduction: One of the most recently disclosed Vitro techniques is based upon the fact that the high-intensity arc can be maintained without vaporizing all of the material in the anode. By manipulating the arc current, the temperature can be kept just below the boiling point of the film of molten metal, carbide, or other reaction product which may be formed on the face of a consumable electrode composed of carbon and

a reducible compound. Under these circumstances, the molten material drips off steadily in the form of spherical globules which freeze before reaching the bottom of the chamber. Volatile impurities can be purged from the molten film even more completely than from the melt in an ordinary vacuum-arc furnace, since they pass out with the carbon monoxide in the tail flame.

Under investigation by Vitro is a variant of this technique involving selective carbon reduction of silicate ores in the liquid phase. Thermodynamic calculations, which are probably more dependable at these lower temperatures than at temperatures of ordinary plasma flames, indicate that rhodonite, for example, instead of being completely volatilized as in earlier laboratory and pilot-plant work, could be treated to produce a tail flame composed of manganese vapor and carbon monoxide, while molten silica plus minor amounts of alkaline earth impurities, would drip off in droplets of molten slag having negligible vapor pressure. This affords an advance compared with the original dissociation process, but it still does not solve the problem of capturing manganese as pure metal after being in the vapor state.

Potential processes

Very little information is available for publication regarding the substantial amount of research conducted outside of the Vitro Laboratories. It is known that work is being done with hot hydrogen for the reduction of metal oxides, especially WO_3. One method is simply to pass the oxide through a heated hydrogen plasma. In view of the well-known water-gas reaction and abundant thermodynamic evidence as well as past experience, hydrogen is classified as a much less active reducing agent than carbon, but if we knew more about chemical kinetics and mass action at plasma temperatures, the capabilities of dissociated and activated atomic hydrogen or of superheated hydrocarbon gases might prove more significant.

Possible metallurgical applications of plasma devices are not confined to the production of reactions in the plasma jet or tail flame. Plasma torches may find employment as heat sources to provide higher temperatures and/or better atmospheric environments in conventional smelting or refining furnaces. Heat transfer rates of a plasma jet are six to eight times those of an oxygen-fed flame. The total heat content includes, not only the sensible heat of the gas at these very high temperatures, but also the heat of dissociation and/or ionization. But owing to the much greater energy content per molecule of gas, the heat removed on contact with a target does not reduce the temperature so rapidly. This means that the molecules next to the target surface need not be replaced as rapidly in order to maintain the desired temperature level. If desired, the high kinetic energy of the gas stream may be employed to propel products rapidly out of the reaction zone or may be recovered in the form of heat.

To those willing to investigate possible uses of plasma devices in extractive metallurgy, perhaps the most obvious suggestion would be to study volatilization processes which would require higher temperatures than are readily available in contemporary Waelz kilns or shaft furnaces, including those used in the various electrothermic processes for zinc. Of course, there is the possibility of devising liquid-phase treatments of high-melting-point metals. In either of these types of approach, the carbon-free feature of the flame might prove advantageous.

Ordinarily, nitrogen or hydrogen may be considered to be the most economical gas to use with a plasma jet. Where these inexpensive gases are incompatible with the process, argon, helium, or any other more suitable gas may be employed. Plasmas from diatomic gases have more heat per atom than those of monatomic gases at the same temperature due to the heat of dissociation; however, the peak arc temperature available in a given torch may be considerably higher in the case of a monoatomic gas. The latter characteristic has no significance in extractive metallurgy, because all plasma temperatures are likely to be considerably higher than can conceivably be needed. Heat contents of 140,000 Btu per lb may be considered typical of the 15,000°F flame issuing from a hydrogen plasma jet. This compares with an enthalpy of about 5000 Btu per lb for an oxygen-acetylene flame at 5600°F.

When the desired end product is a single synthetic compound, it has been convenient to aspirate gases or powders into the flame. This immediately calls to mind possible fluidized bed types of treatment. Such types of operation should be explored, but in cases where slag and metal or any multiple product operation is conceived, the problems of separating ultrafine products and of avoiding agglomeration must be considered.

Shaft-furnace operations would seem to afford an interesting field of speculation. For example, several plasma jets might be injected through the tuyere openings of a conventional blast furnace. The composition of the plasma flame could be reducing, oxidizing, or inert, depending upon the character of the charge, and it might even be varied to suit conditions that might develop temporarily within the smelting column. Inasmuch as heat no longer would have to be generated by supplying oxygen to a bed of incandescent coke, the volume of *blast* would be correspondingly reduced. To minimize radiation losses and to avoid troubles with refractory linings, it would be necessary to confine the reactions to a central core of activity surrounded by unfused charge material. Various other minor changes would be indicated, but after eliminating top temperature barriers, the metallurgist's imagination might be fed by such fantastic ideas as removing silica in the fume from the top of the stack, recovering it as a valuable product, and/or tapping liquid tungsten metal or a refractory carbide from the hearth.

Less adventurous concepts would include the possibilities of using plasma jets merely as temperature *boosters* in an open-hearth, reverberatory, or rotary kiln However, the purpose of this paper is merely to suggest that, since plasma has been identified as the fourth state of matter, it warrants even more active consideration in extractive metallurgy as well as in other branches of science and technology.

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Titanium Founders Day

by R. I. Jaffee, Battelle Memorial Institute

Before memories dimmed, the men active in the introduction of titanium as a defense material met in Washington at the Park Sheraton Hotel on July 18 and 19 to see what conclusions they might draw. The session was called, *Titanium Founders Day*, and the men participating were therefore called *founders*, which was the uninspired residuum from a list of discarded names like *old timers, early birds*, and *didus ineptus—the dodo bird*. The latter is defined by Webster as an extinct bird related to the pigeon but larger than a turkey, which led N. E. Promisel to remark that the purpose of the day perhaps was to determine, "How big a turkey was titanium?"

The session was organized by N. E. Promisel of the Bureau of Naval Weapons, W. J. Harris, Jr., of the Materials Advisory Board, and Colonel John Dick, formerly of the Air Force and currently with Allegheny-Ludlum. Assistance in planning was provided by Dr. Walter Finlay of Crucible Steel and T. W. Lippert of Titanium Metal Corp. of America. Ably staffing Founders Day was Ray Watercotte, on loan from TMCA.

Invitations were sent to about 100 *founders*, most of whom came. Dinner on July 18 was for reacquaintance, gemutlichkeit, and auld lang syne. After dinner, a movie, reputed to have been edited by Ray Watercotte, entitled, *The March of Titanium*, was presented. Most of the replete founders groaned inwardly at the introduction in the style of *The March of Time*—roar of airplanes, tossing of ocean waves, and crash of artillery. Later they were delighted by the context made up of clips from old W. C. Fields comedies, Perils of Pauline, Frankenstein, and Dracula movies, accompanied by a tongue-in-cheek dialogue relating the action on the screen to titanium past history. Also reputed to have been prepared by the ubiquitous Watercotte was a historical brochure entitled *Titanium, Part Product, Part Cause*. This *casual history—lightly salted* described the detailed endeavors of the titanium founders, drawing liberally on documentation from past records. It recounted the soaring forecasts for titanium use from the Malone Committee hearings. The support of users like Pratt and Whitney, who provided the backbone use for titanium, was brought out. More titanium was used in the Pratt and Whitney J-57 than any other application. Pratt and Whitney is now doing pioneering work on titanium in the Pershing motor case. Also cited was the example of General Electric. After dropping titanium for its J-79, G.E. recently appears to be back with titanium, because of four things: performance, low cost, high reliability, and growth potential. Retold too were the booming days for titanium in 1956 when it was being groomed as the prime material for manned military aircraft, prior to the letdown when military policy switched to missiles. The early support of the Army Ordnance was cited, ranging from the early, timely boost in research and development and the well-publicized mortar baseplate to the recent sophisticated application of titanium in the Davy Crockett weapons system, where titanium alloys with a strength-weight capability of 1 million psi are being used for the recoilless rifle and the ammunition-launching pistons.

The main business of Founders Day took place on July 19, *Retro-, Circum-, and Introspection Day*. N. E. Promisel, perhaps the chief materials innovator of the military services, was chairman. He pointed out that titanium development was dedicated to military production and subject to accelerating pressures. Titanium evoked both industrial and Government support, and led to an interplay between Government and industry without precedent.

The founders addressed themselves to four problems: (1) how to introduce a new material; (2) how to deal with critical problems; (3) how to forecast requirements; and (4) how to provide support.

Introducing New Materials

What are the factors that should be taken into account in the timing of the introduction of a new alloy? Should there always be the type of screening reflected by the Titanium Sheet Rolling Program or should the more free-wheeling competitive sales approach be emphasized? Speakers were: W. W. Minkler, TMCA; and Paul Maynard, North American Aviation, Inc., Columbus, Ohio. Commentators were: L. S. Busch, The Brush Beryllium Company; B. S. Mesick, University of Arizona; and S. V. Arnold of Watertown Arsenal.

The large-scale sheet rolling type of program was pointed out as a logical extension of prior Government support of production and capital equipment. Such a program will provide the basis for the further stage of development to the ultimate products. The free-wheeling sales and customer purchasing approach was said to be best introduced after a specific weapons system has been selected. Heat-treated forgings were cited as an example of the application best suited for the direct free-wheeling sales approach. They are relatively simple products to produce, and their development might have been slowed considerably had a major Government program been instituted. A danger inherent in Department of Defense-type programs is that publicity is given to the early difficulties inevitably associated with such a program and that defects not inherent in the product are overemphasized.

From the user's viewpoint, they tend to be more conscious of faults than advantages in early material. The aircraft industry had many tribulations during introduction of titanium sheet. A reason to favor the DOD rather than the sales approach is to protect against the inherent optimism of producers and users in relation to time, costs, and manufacturing difficulties. It is vital to provide design data as well as material. A sheet rolling program was thought not to be always necessary, but it is well to have some sort of screening of new materials, and information-dissemination groups like Defense Materials Information Center brought in.

Dealing With Critical Problems

Recognizing that any new material is going to be plagued by unexpected difficulties, to what extent should extraordinary efforts be made to point out the potential problems in deal-

ing with new material in its earliest development stage? What are the most useful ways to cope with problems of great technical difficulty as they arise, in terms of organization and administration and the dissemination of information. Speaking on this subject were: W. L. Finlay, Crucible; and S. A. Herres of TMCA. Prepared comments were given by R. I. Jaffee of Battelle and W. H. Sharp of Pratt and Whitney.

It was pointed out that when unexpected difficulties occur, they should be examined critically and not rationalized out of existence. During early production days many of the companies were using old equipment, not capable of aircraft quality material. Sheet and mill products were made, but not evaluated carefully. Inadequate equipment accounted for many of the difficulties in the early 50's. It was recommended that first the material be compared with alternate materials using a paper study similar to that used by RAND on beryllium. Next, realistic data related to the intended applications should be obtained. Alloys should be screened by small-scale laboratory ingots, then switched to pilot-size ingots of the order of 10 cubic inches, and the fabrication parameters determined on instrumented mills. On such a scale, process development parameters such as cold work versus hot work, canning versus bare, reduction per pass and over-all, and detailed mechanical tests should be conducted. At this stage the ingots should be sent to independent research laboratories to be examined critically. Detailed, advanced production studies should be coordinated by ad hoc panels such as followed the sheet rolling program.

In dealing with problem areas, foreseeable and unforeseeable must be considered. Government support was recommended on basic materials problems that can be foreseen. Industry is better able to handle simple problems, such as sponge hardness, on a routine research and development basis. Also, problems like exploding furnaces are best handled directly by industry. Unexpected major problems, such as salt-stress corrosion, require Government support, cooperative work between the producers, and symposia such as the one held in conjunction with TML.

In dealing with difficulties, the first method is the use of research and development. The anticipatory or ad hoc type was thought to be best. Post hoc research and development inevitably is more extensive and costs more. The second general method of dealing with problems is not to solve the immediate problem but to solve the more basic one and eliminate the current crisis. Vacuum-consumable-electrode melting, which was developed at about the time of the hydrogen crisis, was a major factor in eliminating this problem in future material. The third way to deal with problems is through utilization of communications channels. Symposia directed at problem areas generally are more rewarding than general symposia aimed at the state of the art. Too many of the latter and too few of the former seem to be held at present. The fourth solution is the large integrated research and development program. The DOD titanium alloy sheet rolling program is a good example wherein the solution of a difficult problem, such as solution treatment of heat-treatable titanium sheet, was vastly speeded up by the program's competitive and feedback feature.

Forecasting Requirements

How can government and industry together establish the technical basis required for continued development for a new material for industry only? How can forecasts with valid predictions be made on a long-term basis? Speakers were Mr. S. N. Norwood, Union Carbide Metals Corp., and F. H. Vandenburg, Oregon Metallurgical Corp.; commentators were Col. John Dick of Allegheny-Ludlum Steel and T. E. Perry of Republic Steel.

It was pointed out that valid forecasts are difficult, even impossible, with the changing base characteristic of military applications. More realism on needs and requirements for new materials was urged. Under circumstances of a changing base, Government support is essential. Industry cannot take the risk. It is also desirable to avoid crash-type programs. Where large-scale development work is necessary, cooperative Government-industry programs are needed. In setting up such programs, attention should be paid to requirements, appraisal of competitive materials, and the capabilities of the industry.

A common theme was the need for improved coordination and communication in establishing technical requirements. Technical information is needed on properties, producibility, and price. Industry and Government need to work closely and realism is needed on both sides. To predict changes in the base of operation, a standing industry-Government committee was thought to be helpful. Forecasting of requirements should be done by an integrated group, not solely by producers or by Government, thereby avoiding the safety factors each tends to put in their estimates.

Providing Support

What are the more useful incentives to encourage orderly and rapid development of high quality products? What is the relative importance of these incentives: research and development contracts, tax amortization, purchase contracts, support to the end-item contractors? Speaking on the subject were A. B. Kinzel of Union Carbide, C. I. Bradford of Du Pont, and T. W. Lippert of TMCA; commenting was W. N. Lawrence of the Department of Defense.

Timing and timeliness were said to be prime considerations in determining the best means of support. Some things may be done during wartime that cannot be done in peacetime. The titanium program was tied up with a war, although a somewhat small one, Korea. Lack of judgment, self-deception, and wishful thinking are the chief faults to be guarded against. Crash programs are not justified. A healthy program should feed on itself, with Government-supported research and development programs to provide information on the possible uses of the metals.

All means for subsidization were used in the forced development of titanium, which resulted in a new industry. Any fault lay in the disappearance of the tonnage market. There probably will be similar forced development of other metals. Of the various methods of subsidy, the most effective was thought to be support for end-item contractors. Purchase contracts were somewhat artificial, while research and development contracting will continue to be used. The main problems in the titanium industry were said to be overcapacity and pressure to reduce prices.

Round Table

Promisel summarized his impressions from the discussion. Some of these were:

(1) Combined Government-industry effort seems to be needed in the low-tonnage, special application, new metal field.

(2) Many problems will emerge which need to be examined on a systematic, long-range basis, without setting up crash programs.

(3) The changing nature of technical requirements should be recognized, and factored into any major program.

(4) In making valid forecasts, a combination is needed of analysis of technical requirements, unit requirements, Government-industry cooperation, and a group for estimating requirements.

(5) Timing is important in justifying the risk of the effort in supporting the new metal.

The session was concluded with the thought that the titanium effort was a unique experience and that Titanium Founders Day will be very helpful in future planning. What happened in titanium will continue to influence the future. What happened at Founders Day in Washington on July 18 and 19, 1960, also would have an impact on future individual, corporate, and Government action.

AIME STAFF:
SECRETARY—ERNEST KIRKENDALL
ASST. SECRETARIES—J. B. ALFORD, H. N. APPLETON, J. C. FOX, R. W. SHEARMAN, C. J. HICKS
ASST. TREASURER—JOHN LYNCH
METALLURGICAL SOCIETY STAFF:
SECRETARY—R. W. SHEARMAN
ASST. SECRETARY—C. C. JOHNSTON

NEWS OF YOUR PROFESSION

Mechanical Conference Set For Pittsburgh January 18, 1961

Seven important subjects will be covered by papers and discussions at the Third Mechanical Working Committee Conference in the Penn-Sheraton Hotel, Jan. 18, 1961.

The conference will be sponsored by The Metallurgical Society's Iron and Steel Division and the Pittsburgh section of AIME.

The general theme of the conference will be *Bar and Allied Products*, with papers and discussions taking up *Control of Bar Mill Quality, Billet and Bar Quality as Affected by Double Conversion and Direct Rolling Practice, Bloom and Billet Conditioning*, and *Effect of Composition in Causing Surface Breaks During Blooming*. Also to be considered are *Extrusion of Steel, Ultrasonic Inspection for Internal Soundness of Semi-Finished Products*, and *Use of Magnetic Particle Testing for Surface Defects on Bars and Allied Products*.

The luncheon speaker, Robert B. Pease, executive director of the Pittsburgh Redevelopment Authority, will discuss the Pittsburgh Renaissance.

The conference proceedings will be published by Interscience Publishers, Inc. of New York.

Call for 1961 Dues

Notice is hereby given that dues for the year 1961 are payable Jan. 1, 1961 as follows: Members and Associate Members, $20; Junior Members for the first six years of Junior Membership, $12; and thereafter $17; Student Members (including an annual subscription to a monthly journal), $4.50.

Dues bills were mailed in early November. Prompt payment will assure uninterrupted receipt of the publications desired in 1961. If, for any reason, a bill is not received within a reasonable time, AIME headquarters should be notified.

AIME Annual Meeting Will Feature Presentation of 1961 Awards

Conferring of awards is an important part of the AIME Annual Meeting each year. The following honors will be awarded during the meeting in St. Louis, Mo., February 26-March 2:

Robert H. Richards Award for 1961—to Nathaniel Arbiter, professor of mining engineering in the Henry Krumb School of Mines, Columbia University, "for his contributions to the mineral industries and to the advancement of our profession, as an educator, as a research scientist, and as an engineer. His ability to analyze and solve difficult problems and his willingness to assist others has brought to AIME and the mineral industry of the world, advancements in flotation and other beneficiation processes."

Robert H. Hunt Award for 1961 to Y. Doi and K. Kasai for their paper, *The Making of Self-Fluxing Sinter and the Blast Furnace Practice with its One Hundred Percent Sinter Burden*, published in JOURNAL OF METALS, November, 1959.

J. E. Johnson, Jr. award for 1961—to Richard J. Wilson, of Chicago, assistant superintendent, plant No. 2 div., Iron Production, Inland Steel Co. Mr. Wilson was selected "for his outstanding contribution to the literature pertaining to the art of blast furnace smelting; and for his untiring efforts to disseminate the information gathered by his own pioneering for the benefit of the industry." The Iron and Steel Div. each year bestows the award on a metallurgist, under 40 years of age, who makes a significant contribution to the manufacture of pig iron.

William Lawrence Saunders Gold Medal Award for 1961—to Marcus David Banghart, vice president of Newmont Mining Corp. "for the development of new mining techniques and overcoming many operating difficulties, resulting in O'okeip and Tsumeb becoming low cost producers under his capable management." Established in 1927, through a gift by Mr. Saunders, a former President of AIME, the award recognizes distinguished achievement in mining other than coal.

(Continued on page 63)

Business Meeting

The regular Annual Business Meeting of the AIME will be held on Tuesday afternoon, February 28 at 4 p.m. in the Khorassan C Room of the Chase Hotel in St. Louis, Mo. The meeting will immediately follow the All-Institute Session at the Annual Meeting.

Electrons In Metals Will Be Discussed At Annual Meeting

A series of three lectures on the behavior of electrons in metals will be featured at the AIME Annual Meeting in St. Louis. The lectures have been arranged by the Chemistry and Physics of Metals Committee, and will be presented in the Old English Room of the Ambassador Hotel on Monday morning, February 26.

It is now possible to probe crystals with ultrasonic beams and thereby measure the shapes of their Fermi surfaces in detail. This will be discussed by R. W. Morse in his talk on *Measurements of the Shapes of Fermi Surfaces in Metals*. The hypothesis that superconductivity in metals results when an energy gap opens up at low temperatures has been confirmed through tunneling experiments. I. Giaver will discuss *Electron Tunneling in Superconducting Metals*. W. B. Pearson will speak on *Thermoelectricity of Metals*. Interest in thermoelectricity is expanding because of its technical applications and because of the insight that studies of it give to the electronic structures of metals.

15th Annual Off-the-Record Meeting Is Held In Pittsburgh on November 4

An Old Timers' Luncheon honored the past chairmen of the Pittsburgh Section during the Off-the-Record Meeting which was held November 4. Back row (l to r), are C. L. McCabe, L. Thiessen, E. H. Hollingsworth, and G. Donaldson. Front row: N. K. Flint, Joseph L. Gillson, President of AIME, who made the presentations, E. H. Dix, R. D. Snouffer, and J. M. Vonfeld.

Perhaps the most diversified program presented on a sectional level, the 15th Annual Off-the-Record Meeting of the Pittsburgh Section of AIME, was held at the Penn-Sheraton Hotel on November 4. The technical sessions represented the coal, steel, nonferrous metals, petroleum, and basic minerals industries.

Because an advance program was not made available to the JOURNAL OF METALS, the proceedings of the more pertinent sessions are outlined in this brief article.

Operating Practices Using Oxygen was the subject discussed by the Pittsburgh Section of the National Open Hearth Committee during the operating session. Papers submitted by J. Cain, D. B. Casley, F. C. Owens, C. H. Ow, Jr., R. Boyle, T. B. Winkler, and D. F. Brion were concerned primarily with the efficiency of oxygen injection, and the effects of oxygen on the furnace operation with regard to charge-to-tap times. Moving over to the other side of the furnace, *Pit Side Quality in the Open Hearth* was discussed by G. Harry, J. F. Lynch, J. J. Oravec, and R. H. Rigdon during the metallurgical session. These papers were concerned with pouring rates, deoxidation practices, and pit-side refractories. During the refractories session, *Basic Roof Design—Classification* was presented by W. S. Debenham and *Basic Roof Design—Performance and Maintenance* was discussed by J. Cerroni, J. R. Todd, C. M. Burks, C. W. Wood, J. Hazel, R. C. Oswald, and M. Sample. A *Review of Basic Oxygen Furnace Linings and Performance* was presented by W. D. Jones. *Application of an Orifice Washer to Open Hearth Flue Gas* by W. M. Bloom was the topic of the combustion session.

The Section's Institute of Metals Group discussed *Magnetic Materials* which included both ferrous and nonferrous metals. Subjects discussed were: *Secondary Recrystallization in Singly-Oriented Silicon Iron*, T. V. Phillip and R. E. Lenhart; *The Microstructure of Low-Carbon 3.25 pct Silicon Steel*, W. C. Leslie, R. L. Rickett, C. P. Stroble, and G. Konoval; *Magnetic Properties Vs Compositional Variation in Supermalloy Produced by Powder Metallurgy*, P. Cohen; and *Manganese-Aluminum Permanent Magnets*, R. C. Campbell and C. A. Julian.

In the afternoon session, *Chemical Purity and Properties* was the topic of discussion. Papers presented were: *Preparation of Ultra High-Purity Metals, Achieving Purity and Homogeneity*, B. F. Oliver; *On Some Mechanical Properties of Zone-Refined Iron*, J. T. Michalak and H. W. Paxton; *The Growth of Oxide Films on Tin*, W. E. Boggs; *Some Effects of Interstitial Elements on Properties of Pure Columbium*, F. R. Cortes and A. L. Field, Jr.; and *The Effect of Interstitial Elements on the Recrystallization Characteristics of Columbium and Columbium Binary Alloys*, R. T. Begley.

The Section's Mineral Industry group presented various phases of extractive metallurgy. *Reactions Between Carbon Potlinings and Hall Bath*, by M. B. Dell, illustrated the effects of various vapors on the deterioration of potlinings. A *Review*

(Continued on page 62)

William A. Wissler Receives Tone Medal At Niagara Meeting

William A. Wissler, retired Union Carbide Metals Co. metallurgist, was awarded the Frank J. Tone Medal of the AIME Niagara Frontier section on November 16 at the Fifth Annual Award Dinner. The award is made annually to a resident of the Niagara Frontier area in recognition of outstanding achievements to the fields of metallurgy and related sciences.

Mr. Wissler has made many important contributions to metallurgical science in the development of alloys for cutting tools and for hard-facing and high-temperature applications. He was the first metallurgist to work with Elwood Haynes in the Haynes Stellite Co., where he developed electric-furnace procedures for making Haynes Stellite cobalt-base alloys. Through their use in cutting tools, American productivity in World War I was greatly increased. When Haynes Stellite Co. was acquired by the Union Carbide & Carbon Corp. in 1921, Mr. Wissler became associated

William A. Wissler, right, receives the 1960 Frank J. Tone Medal award from Charles H. Emery, chairman of the Niagara Frontier section, AIME.

with that firm and remained active in their research efforts until his retirement in 1958.

Throughout his career, Mr. Wissler contributed substantial improvements to materials produced by the hard-facing industry, particularly the cobalt-base alloys. Combining his knowledge of hard-facing materials with his mechanical talent during World War II, he developed automatic machines for the hard-facing of aircraft engine valves. This technology is now employed throughout modern industry. Early in World War II, Mr. Wissler was instrumental in the development of an alloy for an improved barrel liner for machine guns that gave a life ten to fifteen times greater than the previously used steel barrels.

(Continued on page 63)

NSF Grants $20,000 To Metallurgical Society For Transactions Aid

The National Science Foundation has recently granted The Metallurgical Society $20,000 for partial support of TRANSACTIONS OF THE METALLURGICAL SOCIETY OF AIME. The grant is to be used exclusively for reduction of the backlog of approved papers awaiting publication, and will supplement the income that the Society can normally budget for the publication of TRANSACTIONS. The term of the grant is for one year.

During the past two years, the National Science Foundation has been of considerable help in bringing foreign speakers to technical conferences under the sponsorship of The Metallurgical Society, and for publication of scientific material. A list of the grants follows:

1) Annual Meeting, San Francisco, February, 1959—Symposium on *Direct Observation of Dislocations* $500

2) Technical Conference on *Physical Metallurgy of Stress Corrosion Fracture*, Pittsburgh, April, 1959 ..... $2,500

3) International Conference on *Physical Chemistry of Process Metallurgy*, Pittsburgh, April, 1959 ..... $3,500

4) First International Conference on *Powder Metallurgy*, New York, June, 1960 ..... $3,000

5) Technical Conference on *Response of Metals to High-Velocity Deformation*, Estes Park, Colo., July, 1960 ..... $3,500

6) Fall Meeting, Philadelphia, October, 1960—Symposium on *Decomposition of Austenite by Diffusional Processes* $2,270

7) Publication and distribution of booklet, *Careers in Metallurgy and Metallurgical Engineering* $7,500

8) TRANSACTIONS OF THE METALLURGICAL SOCIETY OF AIME—reduction of backlog $20,000

To date, The Metallurgical Society has used $11,148 of grants from the National Science Foundation. In every case of completed projects, the unused portion of the grant has been returned to the National Science Foundation.

Grateful appreciation is due the National Science Foundation for their assistance to The Metallurgical Society in sponsoring projects furthering the aim of better communication of progress in the research and engineering of metals.

RWS

Personals

(Continued from page 4)

Storage Battery Co. of Toledo, Ohio. He was formerly director of engineering and research for Bunting Brass & Bronze Co.

Horace F. Silliman has been named laboratory manager of the Anaconda Co. He will administer personnel policy within the metallurgical department.

Robert Thompson, chief metallurgist of Dominion Engineering Works, Ltd., of Montreal, has received the Award of Merit of the Steel Castings Institute of Canada.

Allen D. Zumbrunnen has been appointed an assistant metallurgical engineer in the Metallurgy div. of Argonne National Laboratory. He will assist in developing new materials for atomic reactors and new methods of fabricating nuclear fuel elements.

R. H. Cutting, vice president of the Bunker Hill Co., will head the company's newly formed marketing dept. **M. E. Elmore,** formerly sales manager of the chemical products div., has been named Pacific div. sales manager, and **A. F. Kroll** has been appointed Pacific div. production manager of the fabricated products div.

Willis F. Thompson Is New President of UET

Willis F. Thompson, of New Haven Conn., has been elected president of United Engineering Trustees, Inc., an organization of five major national engineering societies. Mr. Thompson, who is executive vice president of Westcott & Mapes, Inc., and a former vice president of ASME, succeeds Andrew Fletcher, of New York, chairman of the board of directors of St. Joseph Lead Co., and former president of AIME.

Mr. Thompson and Mr. Fletcher are exchanging posts. Formerly, Mr. Thompson has been chairman of the UET Real Estate Committee, a function now undertaken by Mr. Fletcher.

UET also announced the election of two new vice presidents and two new members of the Board of Trustees. The new UET vice presidents are James F. Fairman, senior vice president of Consolidated Edison Co. of New York, representing AIEE, and L. C. Kemp, Jr., vice president of Texaco, Inc., representing AICE.

The new trustees are Michael L. Haider, vice president and director of Standard Oil Co. of New Jersey, representing AIME, and William H. Wisley, executive secretary of the American Society of Civil Engineers.

Dr. Fies Gets AIME 50-Year Pin

Southeast section, AIME, met November 10, in Birmingham, Ala., honoring Dr. Milton H. Fies, who received a certificate and 50-year pin for service with AIME. Presenting the award to Dr. Fies was E. P. Reed, Southeast section chairman. Dr. Joseph L. Gillson, President of AIME was the principal speaker. He discussed an archeological trip to Mexico and showed color slides. Dr. Fies, an engineer with Alabama Power Co., is a pioneer in the field of underground gasification, attempting to devise a system for burning coal underground, saving cost of extraction, and converting it into power on the scene.

AROUND THE SECTIONS

Pictured at a recent meeting of the Coeur d'Alene subsection in Kellogg, Idaho, are (l to r), A. Y. Bethune, chairman of the subsection, Douglas Baker, secretary-treasurer, C. J. Hicks, western field secretary, AIME, and Carlton C. Long, President of The Metallurgical Society. Dr. Long, the principal speaker, discussed the background and the future of the lead and zinc industry.

New York Section held its annual President's Night meeting on December 7. *Industrial Minerals Used by the Chemical Industry* was the topic of a talk by Joseph L. Gillson, President of AIME. Dr. Gillson described the production of organic chemicals from coal and from natural gas. He also detailed the many inorganic chemicals which are derived from industrial minerals and used by the chemical industry.

Morenci sub-section, AIME, met November 1, at the Longfellow Inn in Morenci, Ariz. Leonard Klein, research engineer of the Phelps Dodge Corp., assisted by John H. Davis, Jr., chief mechanical engineer of the Phelps Dodge Western Engineering Office, presented a program on the *Air Reforming of Natural Gas for Use in the Copper Industry*. This process has been installed in the Douglas Smelter and is now being installed in smelters in Ajo and Morenci. It represents a major advance in copper smelting and fire refining practices and is the result of many years of previous consideration and of many plant tests conducted at the Douglas Reduction Works.

Chicago section, AIME, held a dinner meeting November 2, at the Chicago Bar Association. Neele E. Stearns, director of the executive program, at the University of Chicago, spoke on *The Education of Engineers for Management*.

Nevada section, AIME, will sponsor the *Southwest Mineral Industry Conference* which will be held at the Stardust Hotel in Las Vegas April 24-25, 1961. The Southern California and San Francisco sections of AIME will participate in the conference which will be a joint meeting with the Industrial Minerals div. of SME. The theme of the conference will be *Metals and Mineral Products Meet the Challenge*. After a general meeting, there will be eleven technical sessions at which forty-two papers will be presented. They will cover such subjects as industrial minerals, mining, geology, atomic energy, extractive and physical metallurgy. R. R. McNaughton, 1961 President of AIME will be the speaker at the luncheon on April 25.

Maricopa subsection (Arizona) held a dinner meeting December 1 at the Westward Ho Hotel in Phoenix. The guest speaker was Jack Neal, general manager of the Metate Asbestos Co., who discussed *Mining and Milling Asbestos in Arizona*.

At the November 3 meeting, G. Robert Prescott, research engineer for C. F. Braun & Co., was the guest speaker. He described some of the more severe materials problems in the petroleum industry, and discussed the potential application of some Fe-Cr-Al alloys. The talk was illustrated with slides.

San Francisco section, AIME, held its October 19 meeting at the Engineers' Club with 88 members and guests attending. C. J. Meyer spoke on his recent trip to Africa and discussed the geology of copper deposits in that country.

The section's annual joint meeting with the Student Chapters of Stanford University and the University of California was held November 9 at Berkeley with the University of California Student Chapter as hosts. After an open house at the Hearst Mining Building on the campus, dinner was served at Spengers Fish Grotto. The program, prepared by the University students, included a talk on *Specific Heat of Liquid Bismuth* by Howard Bell, and a discussion of *Dislocation Structures of Molybdenum* by Ray Benson.

Connecticut section held a dinner meeting at the Sanford Barn in Hamden on November 17. Donald P. Senaphim of the IBM Research Laboratory spoke on *Low Temperature Metallurgy*.

Pittsburgh section, IMD group met December 1, at the Mellon Institute. Peter R. Swann, who is with U.S. Steel Corp.'s Edgar C. Bain Laboratory for Fundamental Research, was the guest speaker. Dr. Swann, a graduate of Cambridge University, discussed *The Electron-Microscopy of Deformed Copper Alloys*.

Central New Mexico section, AIME, will sponsor the *Sixth Annual Uranium Symposium* on May 12, 13, and 14, 1961 in Grants, N. M. Harve Ashby is general chairman of the symposium, and James Greenslade is assistant general chairman.

Black Hills section, AIME, elected officers for the coming year during its fall meeting at the South Dakota School of Mines and Technology in Rapid City. Professor E. H. Oshler, head of the Dept. of Mining at the School of Mines, was elected chairman of the section for 1961. Other officers elected were Joel Waterland, first vice-chairman; Paul H. Anderson, second vice-chairman, and Paul DuMontelle, secretary-treasurer. Four men were elected to the executive board of the section. They are John Broome, Fred Moseley, Harold Webb, and Herbert Weisz.

Following the election of officers, William Laval, professor of geology, presented a geological travelogue on a recent tour of Europe.

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STUDENT NEWS

NEW STUDENT MEMBERS

University of Arizona
Bayles, Arthur A.
Monsegur, Michael J.

Carnegie Institute of Technology
Goldman, Richard M.
Robertson, Wayne M.

Case Institute of Technology
Vargo, Joseph A.

Illinois Institute of Technology
Bartosiewicz, Gordon J.
Sandrock, Gary D.

University of Illinois
Waterstrat, Richard M.

Lafayette College
McCain, Kenneth B.

Massachusetts Institute of Technology
Krock, Richard H.

McGill University
Tiivel, Mati

University of Minnesota
Nord, Douglas B.
Ota, Susumu
Schluter, Robert B.

Missouri School of Mines
Borthayre, Philippe P.
Otten, Peter E.

New York University
Bayles, Byron J.
Swirnow, Alan R.

Ohio State University
Eppich, Robert E.

Pennsylvania State University
Kosin, John E.

University of Pennsylvania
Kossowsky, Ram
Van Den Sype, Jaak S.

Purdue University
Chasteen, Jack W.

Rensselaer Polytechnic Institute
Dromsky, John A.
Goodrich, Robert S., Jr.
Kindlimann, Lynn E.
Konrad, Bruce A.
Lee, Albert P.
Pace, John J.
Udy, James C.

Stanford University
Kinsman, Kenneth R.

University of Tennessee
McCoy, Herbert E., Jr.

University of Utah
Hertel, Neil B.

Washington State University
Granger, Guy G., Jr.
Long, Leo H., Jr.
Zimmerly, Merlin R.

University of Wisconsin
Laurhammer, Gerald E.

University of Michigan

Michigan Metallurgical Societies met December 6 to hear M. J. Sennott of the metallurgical dept., discuss a new program for metallurgical engineers at the university. At the November 29 meeting, Dr. Brockway of the chemistry dept., discussed *Oxidation of Single Crystals of Copper*. Douglas Johnston, assistant secretary of The Metallurgical Society, attended the meeting and gave a short talk on AIME-student relations.

Attending the Illinois Institute of Technology's Metals Club meeting on November 15, were (standing, l to r), L. F. Mondolfo, faculty advisor; R. W. Lobenhofer, president; J. B. Schneider, secretary; F. B. Gazella, vice president; F. M. D'Alessandro, treasurer; and (sitting), O. J. Barnett, the guest speaker.

Student News

The JOURNAL OF METALS is eager to give more complete coverage to the activities of the student chapters at the various colleges and universities. Meeting report forms are available upon request. Student news items and photographs should be sent to the News Editor, JOURNAL OF METALS, 29 West 39 Street, New York 18, N. Y.

Solar Energy Digest

Arizona State University has begun publication of a monthly digest in solar energy for the Defense Department. The SEIC (Solar Energy Information Center) Newsletter publishes information on solar energy as it affects the military services. It also carries advance notices of conferences, meetings, and symposia which include papers on solar energy.

Persons desiring copies of the SEIC Newsletter, should address a request for distribution to: Commanding Officer, U. S. Army Signal Research and Development Laboratory, Fort Monmouth, N. J., ATTN: SIGRA/SL-PS.

Illinois Institute of Technology

The Metals Club at Illinois Institute of Technology met November 15 to hear the second in a series of talks concerning the role of the metallurgical engineer in various branches and specializations of the metals industry. O. J. Barnett of Armour Research Foundation was the speaker. The December 6 meeting featured a talk by G. A. Basta, Jr., of J. T. Ryerson & Sons, Inc. He discussed *The Role of the Metallurgical Engineer on Technical Sales.*

Lafayette College

The John Markle Society of Lafayette College met December 8, at Markle Hall; thirty members were present. After a short business meeting, the Luther Witmer Award for the outstanding senior in metallurgical engineering was presented to Lance A. Davis. This was followed by a film, *The Day Before Tomorrow*, from the US Army Ordnance Corps.

Brooklyn Polytechnic

Polytechnic Institute of Brooklyn met November 29, with 20 members present. Ross Peterson of the Magnaflux Corp., discussed *New Developments in Non-Destructive Testing*, and showed slides on the subject. At the November 15 meeting, members saw a film, *Paths of Steel*, dealing with the activities at the U.S. Steel Research Center.

Buffalo NOHC Features Discussion Of Operating Safety

The Buffalo Section of the National Open Hearth Steel Committee held its 11th Annual Fall Meeting on November 15 at Hotel Statler-Hilton, Buffalo. Attendance totaled 229. Feature attraction was a morning plant tour of the No. 3 Open Hearth Shop of Bethlehem Steel Co.

The afternoon technical session included contributions from all major steel plants in the area covered by the Buffalo Section. *Operating Safety* was the subject of a panel discussion.

Synthetic Bessemer Steel was the title of the Section's award-winning paper by Frank Sadler, Open Hearth Metallurgist, Republic Steel Corp. The effect of sulfide inclusions on machinery was described.

A panel discussion on *Pit Practices* included the following participants: William Redmond, Republic Steel Corp. who spoke on *Proper Teeming;* John Hunt, Dominion Foundries and Steel Co. on *Outside Nozzle Setting;* William McShane, Bethlehem Steel Co., on *Capping Practice on Large Molds;* and George Newton, Steel Co. of Canada, on *Ladle Brick, Stopper Rods, and Sleeves.*

A paper on *Future of Open Hearth Steelmaking* by A. Keith Moore, Superintendent of Open Hearths, Steel Co. of Canada, concluded the technical program. Comparisons were made between the newer oxygen steelmaking processes and the traditional open hearth process, in relation to use of scrap and hot metal, the quality of steel produced, and the capital costs for plant expansion. The open hearth furnace has one major advantage—an almost limitless range in its use of raw materials.

A special feature of the banquet was the presentation of a gift to Harry A. Morlock of General Refractories Co., in appreciation of his long and faithful service to the section. Mr. Morlock recently retired as secretary-Treasurer after serving for seven years.

Responsible for the success of the 1960 Fall Meeting were the hard-working officers. Harry A. Koegler, General Refractories Co., is serving as new section secretary-treasurer. Frank Gregory, assistant superintendent of steel production, Bethlehem Steel Co., Lackawanna, presently vice-chairman, will succeed Mr. Moyer as section chairman.

RWS

DOYLE W. RAUSCH

Sommer Scholarship Is Awarded To Doyle W. Rausch

Doyle W. Rausch, a senior in metallurgical engineering at Ohio State University, has been awarded the NOHC's Alvin H. Sommer Scholarship for the 1960-61 academic year. The scholarship, consisting of a $1000 stipend, is awarded in honor of Alvin H. Sommer, a past chairman of the National Open Hearth Steel Committee, and vice president and general superintendent of Keystone Steel & Wire Co.

A native of Dover, Ohio, Rausch was graduated from Dover High School in 1949. In 1956 Rausch entered Ohio State where he is currently completing the fifth year of the combined (Bach. Met.E. and M.S.) program in the Dept. of Met. Eng.

In addition to the NOHC scholarship, Rausch has received the A.S.M. Scholarship (1957-58), the Mershon National Defense Scholarship (1958-59), the Foundry Educational Foundation Scholarship (1959-60), and the Western Electric Co., Scholarship (1959-60).

The funds for the NOHC Scholarship were donated by J. H. Reighart, president of Sticker Industrial Supply Corp., Cleveland, Ohio.

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Off-the-Record

(Continued from page 58)

of Bureau of Mines Experimental Blast Furnace Operations, by W. Mahan, outlined previous work done, and provided for some future possibilities. *Modern Sintering Plant Instrumentation* by J. Hansen, pointed out that the changeover of sintering operations from an art to a science necessitates the increased use of instrumentation. *An Investigation of Particle Size Determination* was presented by G. Grimes. *Research in Hydraulic Mining* by M. J. Akerman led off the afternoon session. A film illustrated the experimental mining operations being carried out by the Bureau of Mines. *Extractive Metallurgy in the Atomic Age,* F. M. Stephens, Jr., surveyed current research and developments in extractive metallurgy, and contemplated some future possibilities. *New Uses for Oxygen in Steelmaking—For Nonferrous Use?* by E. S. Kurzinski reviewed the new basic oxygen processes, and pointed out possible applications to nonferrous metals. *Recent Legislative Developments Respecting Air Pollution* by W. C. L. Hemeon covered the legal aspects for various locales pertaining to air pollution. A film, *Refining Nickel from the Sudbury Ores,* concluded the session.

Additional sessions of the Coal Division and the Petroleum Sub-Section rounded out the technical program. The Student Affairs Committee of the Section invited some 150 students from neighboring colleges to participate in the special student program. W. H. Meanor, coordinator, personnel procurement of Allegheny Ludlum Steel Corp. spoke on *Your Best Foot Forward* to the student session.

An *Old Timers'* Luncheon honored the past chairmen of the Section with AIME President Joseph L. Gillson making the presentations.

The all-day sessions were followed by a cocktail party made possible through the courtesy of various suppliers. J. R. Rohleder of Bell Telephone Co., guest speaker at the Fellowship Dinner, ably described the US's attempt to put a man into outer space—*Project Mercury.* This thought-provoking talk stimulated discussion from the floor, bringing to a close a very informative day.

A huge vote of thanks and a *job-well-done* should go to the officers and members of the Pittsburgh Section of AIME, and especially to R. G. Redelfs and R. L. Llewellyn, Chairman and Vice-Chairman of the Program Committee, and to the other members of the Committee who made this excellent meeting possible.

EJF

MEMBERSHIP

Proposed for Membership
Metallurgical Society of AIME

Total AIME members on November 30, 1960, was 34,502; in addition 2,704 Student Members were enrolled.

ADMISSIONS COMMITTEE

W. L. Brytczuk, Chairman; S. C. Carapella, Jr.; J. W. Hanley; T. D. Jones; H. C. Larson; Harold Margolin; Shadburn Marshall.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

Members

Acker, M. Worth, Philadelphia, Penn.
Brauns, Frank E., Chicago, Ill.
Burger, Charles G., Hammond, Ind.
Colburn, William A., Denver, Colo.
Cooper, Gordon E. T., Hamilton, Ont., Canada
Crankshaw, Fred, Letchworth Herts, England
Czaruk, Michael A., Chicago, Ill.
Jordan, Charles B., Pasadena, Calif.
Lillys, Peter, Syracuse, N. Y.
McBride, Floyd E., Cleveland, Ohio
McKee, Keith H., St. Clair Shores, Mich.
Noggle, Thomas S., Oak Ridge, Tenn.
Palacios, Jaime L., Copiapo, Chile
Pennington, William A., College Park, Md.
Pikus, Francis W., Cleveland, Ohio
Ryan, James W., Corning, N. Y.
Scharfstein, Laurence R., Reading, Pa.
Singh, Gurdev, Kirkee, Poona-3, India
Solnick, Robert L., Fullerton, Calif.
Soltis, Paul J., Reading, Pa.
Swier, Cazimir V., Glen Cove, N. Y.
Williams, Clarence A., Monessen, Pa.

Associate Members

Dobbener, Raymond, Duisberger, Germany
Fullet, William H., Pittsburgh, Pa.
Mannix, Francis L., Boston, Mass.
Martin, Joseph P., Newfield, N. J.
McIlwain, Samuel D., Louisville, Ky.
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Cooke, Elliott F., Rahway, N. J.
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McIntyre, Edmund H., Regina, Sask., Canada
Milhaljevic, Joseph E., Duquesne, Pa.
Scroggins, Lloyd A., Indiana Harbor, Ind.
Sylvester, Walter R., Aliquippa, Pa.
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REELECTION—MEMBER

Bigley, Arthur C., Jr., Anaconda, Mont.

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Horn, Howard N., Chatham, N. J.

REINSTATEMENT—CHANGE OF STATUS

Junior to Member

Seder, Peter G., Talcahuano, Chile

Student to Member

Perduyn, James A., Garden Grove, Calif.

1961 Awards . . .

(Continued from page 57)

Hal Williams Hardinge Award for 1961—to Raymond B. Ladoo, consulting engineer, author of major technical works, and an outstanding figure in the field of industrial minerals. The designation of Mr. Ladoo was "for his work as engineer and author in bringing about wider recognition of the vital part played by industrial minerals in the national economy." The Hardinge Award was established in 1958 in memory of a noted inventor and manufacturer of mining and milling equipment. It recognizes outstanding achievement in the field of industrial minerals.

Mathewson Gold Medal—to K. T. Aust and J. W. Rutter of the General Electric Research Laboratory for the following papers: *Effect of Solute Impurities on Preferred Orientation in Annealed High-Purity Lead*, TRANSACTIONS Volume 218; *Grain Boundary Migration in High-Purity Lead and Dilute Lead-Tin Alloys*, TRANSACTIONS Volume 215; *Temperature Dependence of Grain Migration in High-Purity Lead Containing Small Additions of Tin*, TRANSACTIONS Volume 215.

Erskine Ramsay Gold Medal Award to Donald Markle—"for his pioneering contributions to the beneficiation and utilization of fine sizes of anthracite and for his life-long leadership in the anthracite industry and in civic activities in the anthracite community in Pennsylvania."

The James Douglas Gold Medal —to Frank A. Spedding "for his imaginative pioneering in the technology associated with early atomic energy activities and his contributions to the extractive metallurgy of the rare earths." Mr. Spedding is director of the Ames Laboratory of the U.S. Atomic Energy Comm., Iowa State Univ., Ames, Iowa.

Extractive Metallurgy Division Award—to H. H. Kellogg and S. K. Basu for their paper on *Thermodynamic Properties of the System Pb-S-O to 1100°K* which appeared in the bimonthly TRANSACTIONS OF THE METALLURGICAL SOCIETY, Volume 218, February, 1960.

The Benjamin F. Fairless Award —to T. E. Millsop, president of the National Steel Corp., Weirton, W. Va.,—"for his accomplishments in behalf of the steel industry, for his support of continuing technical progress in the industry, and for his civic endeavors."

Anthony F. Lucas Gold Medal to Edwin Oliver Bennet—"for fostering the development of gas pressure maintenance in oil reservoirs; supervising the first field stabilization of high gravity crude oil, and developing the technique of multiple zone completion of wells. For his pioneering in the design of high pressure gas cycling plants resulting in increased recovery of condensate from reservoirs."

Rossiter W. Raymond Award for 1961—to Douglas W. Fuerstenau, associate professor of metallurgy, University of California, for his paper, *Retention Time in Continuous Vibratory Ball Milling*, published in MINING ENGINEERING, December, 1959.

Tone Medal . . .

(Continued from page 58)

His achievements earned for him the Army-Navy, the Naval Ordnance, and Office of Scientific Research and Development awards for outstanding technical achievement.

The introduction of the recipient, describing his lifetime devoted to the successful development and application of cobalt-base alloys and to work on tungsten and tungsten-carbide fabrication, was given by Hendrik Dewet Erasmus, senior research consultant of Union Carbide Metals Co.

In accepting the award, Mr. Wissler traced the history of Haynes Stellite cobalt-base alloys. Their superiority to high-speed steels in lathe tools at the high temperature of cutting metal was recognized in World War I production of munitions. Soon the bright reflecting surface of these alloys meant replacement of the easily shattered glass of battleship searchlight reflectors.

Presentation of the Tone Medal was made by Charles H. Emery of Simonds Saw and Steel Co., chairman of the Niagara Frontier section, who served as toastmaster. Joseph L. Gillson, president of AIME, spoke briefly on *News of the AIME*. Greetings from The Metallurgical Society were extended by Robert W. Shearman, secretary. The invocation was given by the Reverend W. Lever Bates, Rector of the Church of the Ascension, Buffalo. The dinner meeting was held at the Stuyvesant Hotel, Buffalo.

The Tone Medal Award was established in 1956 by F. J. Tone, Jr., and Franchot Tone to honor their father, who had made many contributions to the development of silicon carbide and was responsible for the first commercial production of silicon.

NECROLOGY

Year Elected	Name	Date of Death
1913	Gidel, Murl H.	Mar. 1960
1943	Given, Edmond V.	Sept. 7, 1960
1912	Grace, E. G.	July 25, 1960
1915	Greninger, Ira L.	Unknown
1940	Haas, Gilbert G.	Aug. 14, 1960
1926	Hughes, Robert W.	Sept. 1960
1907	Lindberg, C. O. (Legion of Honor)	Sept. 2, 1960
1927	Magee, John F.	Aug. 3, 1960
1920	McDougall, B. W. W.	Feb. 3, 1960
1934	Millard, Frank W.	July 8, 1960
1960	Miller, Robert J.	Unknown
1922	Nobs, F. Wm.	May 21, 1960
1941	Otto, Merle F.	Aug. 4, 1960
1934	Raymond, Robt. L.	Oct. 18, 1960
1932	Robinson, Carel	Sept. 20, 1960
1931	Sachs, George	Oct. 31, 1960
1944	Salmi, Gust J.	Unknown
1957	Sargent, S. B.	Unknown
1928	Shaw, Quincy A.	May 8, 1960
1951	Udin, Harry	Apr. 2, 1960
1943	Volterra, Renzo U.	Mar. 7, 1960
1924	Wagner, H. A.	Apr. 11, 1960

Coming Events

Jan. 8-12, 1961, Symposium on Thermoelectric Energy Conversion, Statler-Hilton Hotel, Dallas, Texas.

Jan. 18, 1961, Cleveland Section, NOHC, Dinner Meeting, Coach House, Strongville, Ohio.

Jan. 18, 1961, Western Section, NOHC, Roger Young Auditorium, Los Angeles.

Jan. 18, 1961, Conference on Bar and Shaped Products, sponsored by Mechanical Working Committee of The Metallurgical Society, Penn-Sheraton Hotel, Pittsburgh.

Feb. 1, 1961, N. Y. Section, Powder Metallurgy Group, Brass Rail Restaurant, 43rd St. & Fifth Ave., New York.

Feb. 10, 1961, Southwestern Section NOHC, Annual All-Day Meeting, Hotel Lennox, St. Louis.

Feb. 26-March 2, 1961, AIME Annual Meeting, Ambassador and Chase-Park Plaza Hotels, St. Louis, Mo.

Mar. 13-14, 1961, 59th Annual Meeting, Steel Founders' Society of America, Drake Hotel, Chicago.

Mar. 20-24, 1961, Western Metal Exposition & Congress, Pan Pacific Auditorium, Los Angeles.

Apr. 10-12, 1961, 44th National Open Hearth Steel Conference and Blast Furnace, Coke Oven, and Raw Materials Conference, Sheraton Hotel, Philadelphia.

Apr. 12-14, 1961, AIME International Symposium on Agglomeration, Sheraton Hotel, Philadelphia.

Apr. 24-25, 1961, AIME Southwest Minerals Industry Conference, Stardust Hotel, Las Vegas, Nev.

Apr. 26-27, 1961, Cleveland Section and The Metallurgical Society, Technical Conference on High Temperature Materials, Hotel Carter, Cleveland.

May 18-19, 1961, AIME 15th New England Regional Conference, Hotel Van Curler, Schenectady, N. Y.

Aug. 30-Sept. 1, 1961, AIME Third Technical Conference on Semiconductors, Ambassador Hotel, Los Angeles.

Oct. 17-Nov. 7, 1961, Visit of the Iron & Steel Institute (United Kingdom) to the United States.

Oct. 23-26, 1961, Fall Meeting of The Metallurgical Society of AIME, Pick-Fort Shelby Hotel, Detroit.

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ANNUAL MEETING — TECHNICAL PROGRAM

The Metallurgical Society of AIME

February 26 to March 2, 1961

Ambassador and Chase-Park Plaza Hotels, St. Louis, Missouri

Condensed Program — Technical Sessions, Ambassador Hotel, St. Louis

	Empire	Coronation	Embassy	Versailles	Marquis
Sunday, Feb. 26 2:30 pm		Symposium on Metallurgical Education p. 66			
Monday, Feb. 27 9:30 am	Metallurgy in the Air Force (IMD) p. 66	Reactive Metals (EMD) p. 66	Copper I (EMD) p. 66	Gases in Steel (ISD) p. 66	Behavior of Electrons in Metals (IMD) p. 67
Monday, Feb. 27 2:30 pm	Metallurgy in the Air Force (IMD) p. 67	Refractory Metals (EMD) p. 67	Copper II (EMD) p. 67	Stainless Steelmaking (ISD) p. 67	Solid-Solution Strengthening and Transformations (IMD) p. 67
Tuesday, Feb. 28 9:00 am 11:00 am	Physical Chemistry of Slags (ISD) p. 69 Howe Memorial Lecture (ISD) p. 71	Application of Carbon in Pyrometallurgy Industry (EMD-MBD) p. 69		Titanium Research (IMD) p. 69	Dislocations (IMD) p. 70
Wednesday, March 1 9:00 am 11:00 am	Recovery and Recrystallization (IMD) p. 72 IMD Lecture p. 75	Beryllium (EMD) p. 73	Hydrometallurgical Treatment of the More Common Metals (EMD) p. 73	Thermodynamics of Liquid Iron (ISD) p. 73	Diffusion and Surface Phenomena (IMD) p. 73
Wednesday, March 1 2:00 pm	Direct Observations of Imperfections in Crystals I (IMD) p. 75	Hydrometallurgical and Other Treatment of Refractory and Rarer Metals (EMD) p. 75	Deformation and Strengthening (IMD) p. 75	Application of Thermodynamics (ISD) p. 77	Deformation of Semiconductors (IMD) p. 77
Thursday, March 2 9:00 am 11:00 am	Ceramics (EMD) p. 78 EMD Lecture p. 83	Direct Observations of Imperfections in Crystals II (IMD) p. 78	Zinc Round Table Discussion (EMD) p. 81	Steelmaking (ISD) p. 81	Structure and Properties (IMD) p. 81
Thursday, March 2 2:00 pm	Refractories (EMD) p. 83	Direct Observations of Imperfections in Crystals III (IMD) p. 83	Extractive Metallurgy of Zinc (EMD) p. 85	Blast Furnace Technology (ISD) p. 86	Phase Relations (IMD) p. 86

Additional Sessions at Chase-Park Plaza Hotel

	Zodiac Room	Starlight Roof	Khorassan C	Tiara
Sunday, Feb. 26 8:00 pm	Council of Education Meeting (AIME) p. 66			
Tuesday, Feb. 28 9:00 am		Chemical Processes (MBD-EMD) p. 71		
Tuesday, Feb. 28 2:30 pm			All-Institute (AIME) p. 71	
Wednesday, March 1 9:00 am		Basic Science Symposium (MBD-EMD-ISD) p. 74		Engineering Management (AIME) p. 73
Wednesday, March 1 2:00 pm		Basic Science Symposium (MBD-EMD-ISD) p. 78		
Thursday, March 2 2:00 pm	Pyrolysis and Agglomeration (MBD-EMD) p. 87			

SUNDAY, FEBRUARY 26

Met Soc — Panel Discussion

Sponsored Research As It Relates to the University, its Function, and Its Faculty

(Arranged by Education Committee of The Metallurgical Society)

2:30 pm — Ambassador Hotel — Coronation Room (Lobby Floor)

Chairmen:
M. E. Nicholson, Jr., University of Minnesota
W. R. Hibbard, Jr., General Electric Co.

Panelists:
The University—M. E. Fine, Northwestern University
The Government—C. F. Yost, Advanced Research Projects Agency
Industry—J. J. Harwood, Ford Motor Co.

Invited Discussions:
Bruce Chalmers, Harvard University
Morris Cohen, Massachusetts Institute of Technology
W. J. Harris, Jr., National Research Council
O. T. Marzke, U.S. Steel Corp.
D. K. Stevens, Atomic Energy Commission

Council of Education — Social Hour

5:00 pm — Chase-Park Plaza Hotel — Zodiac Room

Council of Education — Buffet Dinner

6:00 pm — Chase-Park Plaza Hotel — Starlight Roof

Council of Education — Technical Session

8:00 pm — Chase-Park Plaza Hotel — Zodiac Room

MONDAY, FEBRUARY 27, AM

IMD — Metallurgy in the Air Force—Requirements, Applications, and Research

9:30 am — Ambassador Hotel — Empire Room (Lobby floor)

(Sponsored by the Institute of Metals Division)

Chairmen:
Col. E. M. Douthett, Directorate of Research and Technology
David Swan, Union Carbide Metals Co.

Research and Development and the Future Air Force:
Maj. Gen. V. R. Haugen, Asst. Deputy Chief of Staff, Development.

Basic Research in the Air Force:
Maj. Gen. D. E. Hooks, Air Force Research Division, Air Research and Development Command.

Defining the Environment:
L. F. Salzberg, Physics Laboratory, Wright Air Development Division

Aerospace Structural Materials:
Lt. Col. H. C. Hamlin, Applications Laboratory, Wright Air Development Division

Aerospace Propulsion Materials:
Jerome Krochmal, Materials Central, Wright Air Development Division

EMD — High Temperature Metals (I)—Reactive Metals

(Sponsored by EMD High Temperature Metals Committee)

9:30 am — Ambassador Hotel — Coronation Room (Lobby Floor)

Chairmen:
E. M. Jost, Metals and Controls Division, Texas Instruments, Inc.
R. A. Gustison, Union Carbide Metals Co.

Fused Salt Scrubbing of $ZrCl_4$:
D. R. Spink, Carborundum Metals Co.

Operation of 10,000 ampere Cells for Titanium and Chromium:
W. W. Gullett, Chicago Development Corp.

Na^+-$TiCl_2$-$TiCl_3$ Equilibrium in NaCl Melts:
Alex Boozenny, Titanium Metals Corp. of America

Extractive Metallurgy of High-Temperature Metals:
C. A. Krier, Battelle Memorial Institute.

EMD — Extractive Metallurgy of Copper (I)

(Sponsored by EMD Copper, Nickel, and Precious Metals Committee)

9:30 am — Ambassador Hotel — Embassy Room (Lower Level)

Chairmen:
J. B. Clemmer, U.S. Bureau of Mines.
J. G. Leckie, American Smelting and Refining Co.

Gaseous Reduction of Oxygen-Containing Copper:
Leonard Klein, Phelps Dodge Corp.

Converter Smelting of Copper Using Oxygen-Enriched Air:
Tamon Tsurumoto, Nippon Mining Co., Japan

By-Product Molybdenum Recovery from Western United States Porphyry Copper Ores:
J. A. McAllister, American Cyanamid Co.

Thermal Decomposition of Cobalt Sulfate:
J. S. Warner, Columbia University.

ISD — Gases in Steel

9:30 am — Ambassador Hotel — Versailles Room (First Floor)

Chairmen:
E. C. Rudolphy, U.S. Steel Corp.
J. M. Uys, Allegheny-Ludlum Steel Corp.

Gases in Liquid Steel:
R. J. King, U.S. Steel Corp.

Effect of Hydrogen on Magnetic Properties of High-Purity Iron:
J. F. Nicholson, Wright-Patterson Air Force Base, and R. Speiser, Ohio State University

A Test for Measuring Flake Sensitivity:
J. E. Steiner, U.S. Steel Corp.

Pilot Plant Result of Vacuum Degassing on a 5-Ton Scale:
Marc Allard, Institut de Recherches de la Siderurgie, France.

Solubility of Nitrogen in Liquid Fe-Ti Alloys:
Mohan Rao and N. A. D. Parlee, Purdue University.

IMD

Behavior of Electrons in Metals

(Sponsored by IMD Chemistry and Physics of Metals Committee)

9:30 am Ambassador Hotel Marquis Room (First Floor)

Chairman:
J. J. Gilman, Brown University

Electron Tunneling into Superconducting Metals:
I. Giaver, General Electric Co.

Measurements of the Shapes of Fermi Surfaces in Metals:
R. W. Morse, Brown University

Thermoelectricity of Metals
W. B. Pearson, National Research Council (Canada).

MONDAY, FEBRUARY 27, PM

All-Institute Welcoming Luncheon

12:15 pm Chase-Park Plaza Hotel Khorassan Room
Joseph L. Gillson, President, AIME, Presiding

IMD Metallurgy in the Air Force—Requirements, Applications, and Research

2:30 pm Ambassador Hotel Empire Room (Lobby Floor)

(Sponsored by the Institute of Metals Division)

Chairmen:
Col. E. M. Douthett, Directorate of Research and Technology
David Swan, Union Carbide Metals Co.

Air Force Applied Research Program in Materials:
E. M. Glass, Materials Central, Wright Air Development Division

Role of Solid State Science in Scientific Research:
1st. Lt. R. A. Schalow, Air Force Research Division, Air Research and Development Command

Metallurgy Research at Aeronautical Research Laboratory:
H. A. Lipsitt, Air Force Research Division, Air Research and Development Command

Ceramics Research at Aeronautical Research Laboratory:
Capt. H. Rizzo, Air Force Research Division, Air Research and Development Command

Generation of Ultra High Pressures:
W. G. Field, Air Force Research Division, Air Research and Development Command

Diamond Research:
Capt. Gordon O'Shesky, Air Force Research Division, Air Research and Development Command

EMD

High Temperature Metals (II)—Refractory Metals

(Sponsored by EMD High Temperature Metals Committee)

2:30 pm Ambassador Hotel Coronation Room (Lobby Floor)

Chairmen:
D. H. Baker, Jr., U.S. Bureau of Mines
C. E. Barnett, New Jersey Zinc Co.

Metal Deposition Rates in Filament Bundles:
J. H. Oxley, Battelle Memorial Institute, C. E. Dryden, Ohio State University, J. E. Oberle, and G. H. Kesler, National Steel Corp.

Tungsten Coatings From the Thermal Decomposition of Tungsten Hexabromide:

R. M. Caves, National Aeronautics and Space Administration.

Bomb Reduction of Tungsten:
F. E. Block, P. C. Good, and M. J. Ferrante, U.S. Bureau of Mines.

Oxidation Properties of Molybdenum-Hafnium Alloys:
C. E. Armantrout and H. Kato, U.S. Bureau of Mines.

EMD

Extractive Metallurgy of Copper (II)

(Sponsored by EMD Copper, Nickel and Precious Metals Committee)

2:30 pm Ambassador Hotel Embassy Room (Lower Level)

Chairmen:
C. H. Curtis, Duval Sulphur and Potash Co.
F. L. Holderreed, The Anaconda Co.

Copper Segregation at the Lake Shore Mine:
G. A. Freeman, Transarizona Researches, Inc., and Carl Rampacek and LaMar G. Evans, U.S. Bureau of Mines.

Role of Addition Agents and Purification of Electrolyte with Activated Carbon at the Utah Refinery:
W. M. Tuddenham and H. Lanier, Kennecott Copper Corp.

Role of Bacteria in the Alteration of Sulfide Minerals:
E. E. Malouf and S. R. Zimmerley, Kennecott Copper Corp.

Annealability of Wirebar Copper:
W. M. Tuddenham, and H. Lanier, Kennecott Copper Corp.

ISD Stainless Steelmaking

2:30 pm Ambassador Hotel Versailles Room (First Floor)

Chairmen:
C. W. Sherman, Jones & Laughlin Steel Corp.
G. N. Goller, Armco Steel Corp.

Thermodynamic Properties of CrC at High Temperatures:
S. Fuhishiro and N. A. Gokcen, University of Pennsylvania

C-Cr-Fe Liquidus Surface:
N. R. Griffing, Linde Co., W. D. Forgeng and G. W. Healy, Union Carbide Metals Co.

Equilibrium in the Fe-Si-O System:
R. A. Walsh and S. Ramachandran, Allegheny-Ludlum Steel Corp.

Effect of Tin in Types 302, 410, and 430 Stainless Steel:
E. C. Rudolphy, U.S. Steel Corp.

Improving the Hot Workability of Stainless Steel Containing Lead and Tin:
D. W. P. Lynch, Vanadium Corp. of America.

IMD

Solid-Solution Strengthening and Transformations

(Based on submission of research abstracts)

2:30 pm Ambassador Hotel Marquis Room (First Floor)

Chairmen:
J. B. Cohen, Northwestern University
R. L. Fleischer, General Electric Co.

MONDAY, FEBRUARY 27, PM

Solid Solution Strengthening of Ag by Al:

A. A. Hendrickson, Michigan College of Mining and Technology, and M. E. Fine, Northwestern University.

The CRSS and the strain rate dependence of the flow stress are reported for Ag base Al single crystals containing 1, 2, 3, 4 and 6 atomic pct Al. The strengthening effect of Al additions to Ag depends strongly on the testing temperature: at 415°K, the difference between the CRSS's for 1 and 6 atomic pct Al crystals is 350 gm/mm²; at 4.2°K, this difference is 1400 gm/mm². The strain rate dependence of the flow stress in the easy glide range varies strongly with both the Al content and testing temperature but only slightly with strain; at a given testing temperature, the flow stress dependence on strain rate increases by a factor of about 4 as the Al content is increased from 1 to 6 atomic pct Al.

Observations on the substructure and the effect of crystal growth rate on the CRSS are also given. A model which considers the effect of Al additions on the dislocation density and dislocation width is given to account for the solid solution strengthening.

Solid Solution Strengthening in α Cu-Al Single Crystals:

T. J. Koppenaal and M. E. Fine, Northwestern University.

The critical resolved shear stress of copper base single crystals containing 0.5 to 14 atomic pct Al has been determined from 4.2°K to 523°K. The room temperature CRSS increases from 0.35 kg/mm² for the 0.5 atomic pct Al alloy to 1.55 kg/mm² for the 14 atomic pct Al alloy. Ratios of τ_{77}/τ_{293} were determined and found to be 1.36, 1.84, 1.90, 2.04 and 2.12 for alloys of 0.5, 2.5, 5, 10 and 14 atomic pct Al, respectively. Activation volumes have been calculated from strain rate measurements at 4.2°K and it is noted that the activation volume decreases with increasing Al additions. By use of the activation volume and CRSS data, activation energies for the dislocation cutting process have been calculated; the activation energy increases with increasing Al additions. The results will be discussed in terms of dislocation densities and the relative widths of extended dislocations.

This research was supported by the Office of Naval Research.

Strengthening of LiF Crystals by Magnesium Diffused Surface Regions:

Robert Rosenberg and Irving Cadoff, New York University.

The strengthening of crystals by surface coating has been attributed to both the restriction of the inherent slip processes within the base material and the added mechanical properties of the surface material. To further delineate the mechanisms of strengthening involved, the surface treatment of LiF was investigated.

Initially, magnesium was diffused into the crystal from the vapor at 700°C and the crystal subsequently furnace cooled. A subsequent bend test showed (1) complete embrittlement of the inherently ductile material in that fracture occurred without noticeable macroscopic yielding and (2) the stress at fracture was about an order of magnitude higher than that found in pure LiF (11000 psi vs 1300 psi). Examination under polarized light revealed that the surface region was totally effective in preventing the slip bands produced in the interior from propagating to the surface. Etching confirmed this and showed that the dislocations penetrated the surface region only to a small degree except at the fracture face where complete penetration occurred. Etching also revealed a high density of pits in the surface layer, suggesting some type of precipitate which was subsequently identified by X-ray analysis to be MgF_2. The phase diagram MgF_2: LiF is of a eutectic nature, showing considerable solubility of Mg in LiF at the eutectic temperature and a rapidly decreasing solubility line. To make use of this apparently age hardening system, additional controlled diffusion and aging experiments were carried out. The results are summarized below:

(1) As the temperature of treatment is increased, the rate of penetration of the surface layer into the crystal increases following an Arrhenius relation with an activation energy of approximately 25Kcal/mole.

(2) The increase in depth with time follows the parabolic law ($x^2 = kt$) typical of oxidation-type reactions.

(3) By quenching the specimens from the annealing temperature, one could eliminate the surface pits previously found. This suggests the presence of a supersaturated solution. The rate of strength increase with thickness of the surface region was greater in the quenched samples than in those furnace cooled, indicating loss in strength through macroscopic precipitation.

(4) Aging samples at 300°C after a 600°C anneal and quench treatment produced a transition from brittle to ductile behavior with corresponding decrease in strength. Increased ductility was apparently obtained when the slip bands were able to penetrate through the Mg depleted regions resulting from agglomeration of MgF_2.

(5) Coating with other compounds indicated the necessity of forming a diffusion zone for strengthening since the surface coating itself did not add to the strength.

At least three factors are involved in the strengthening process: (1) strength of the surface region (2) increase in strength of the base LiF and (3) charge effects caused by the formation of positive ion vacancies.

This work was supported by the Office of Naval Research.

Stacking Fault Energy and Work-Hardening of Copper Alloy Crystals:

A. H. King, Arthur D. Little, Inc., and Peter Haasen, University of Göttingen (Germany).

The work-hardening of fcc copper-gallium and copper-germanium alloys has been investigated as a function of composition, temperature, and stain rate. Single crystals of a predetermined orientation were grown from the melt by a modified Bridgeman method, and deformed in tension at temperatures from 78 to 465°K and at strain rates of from 10^{-4} to 10^{-1} per second.

The shear stress at the beginning of cross-slip, τ_0, was found to increase with concentration as would be expected for a decrease in stacking fault energy as the alloy composition approaches the hexagonal phase. The temperature and strain rate dependence of τ_0 were used to calculate γ, the stacking fault energy, which is found to drop rapidly with increasing concentration to a value of 10 ergs/cm² or less at an electron concentration of 1.15 to 1.20. Mechanical twinning was observed in specimens of all compositions, occurring at temperatures up to 200°C in the more concentrated alloys.

The observed work-hardening behavior may be explained in terms of dislocation theory, involving pile-ups of around 50 dislocations at barriers during stage II of the deformation. This hypothesis is supported by electron microscopic observations of slip-steps.

Heat Treatment of Plutonium:

H. R. Gardner and I. B. Mann, General Electric Co.

The heat treatment of plutonium was studied using the Jominy end quenching technique commonly used for determining the hardenability of steel. Two plutonium specimens were end-quenched from temperatures in each of the beta, gamma, delta, delta-prime, and epsilon phases. One series of specimens with a low iron content, 165 ppm Fe, and another group with a high iron content, 678 ppm Fe, were used in order to study the effect of a Pu-Pu_6Fe eutectic network on the hardness and microstructure. Cooling curves were obtained at distances from the quenched end of 1/16, 1/8, 3/8, 3/4, 1-1/4, and 2 in. so that observed effects could be related to cooling rate.

Hardness traverses were made along the length of each of the end quenched specimens. It was determined that only minor variations in hardness were obtained.

Metallographic studies indicated major effects on microstructure. Grain size was found to vary markedly with quenching temperature and cooling rate. The higher the temperature of annealing prior to end quenching the finer the grain size at the quenched end of the Jominy specimen. In addition as cooling rate in a particular Jominy specimen decreased or as distance from the quenched end increased, grain size increased, in some cases by a factor of 25.

In the high iron specimens end quenched from the epsilon phase at 533°C the Pu-Pu_6Fe eutectic present was observed to go into solution and reprecipitate during quenching. The grain size outlined by the eutectic varied markedly with cooling rate, from 0.003 mm at the quenched end to 0.040 at the other end.

An unidentified spherical inclusion was observed to go into solution in the delta and higher temperature phases. Upon quenching the inclusion reprecipitated and its size was effected by cooling rate.

An empirical relationship was established between the grain size of plutonium and the grain size outlined by the Pu-Pu_6Fe eutectic network.

An "Intermediate" Type Habit Plane for the Martensite Transformation in an Fe-Cr-C Alloy:

K. A. Johnson and C. M. Wayman, University of Illinois.

The austenite-martensite transformation in a high purity Fe-3.09 Cr-1.51 C alloy (M_s — 18°C) was studied with particular emphasis on the crystallography of the transformation. Martensite habit planes were determined by means of a two-surface analysis, and the austenite-martensite lattice orientation relationship was obtained using a microbeam Laue technique on a particular plate whose habit plane direction cosines were known.

Four different specimens containing a total of twelve martensite plates were studied. The habit plane poles of these plates when referred to a common unit triangle fell within a cone having a diameter of three degrees. Although irrational, this habit plane can be represented by the approximate Miller indices $(5, 8, 18)_A$, and clearly falls between the $(225)_A$ and $(259)_A$ type habits. Although this habit plane is substantially different, the austenite-martensite orientation relationship was found to be very close to that observed by Greninger and Troiano for an Fe-22 Ni-0.8 C alloy which exhibited a $(3, 10, 15)_A$ habit plane.

The experimental results have been interpreted in the light of present theories on the crystallography of martensite formation. The significance of an irrational shear direction associated with the lattice invariant shear is considered.

This work was sponsored by the Air Force Office of Scientific Research.

Effect of Some Rare Earth Elements on the HCP-BCC Transformation of Zirconium:

L. Ianniello, Argonne National Laboratory, and A. A. Burr, Rensselaer Polytechnic Institute.

The effect of the solutes Nd, Y, Dy, Ho, and Er on the allotropic transformation of Zr has been studied by means of dilatometry. Binary alloys of the above elements with iodide crystal Zr were made by vacuum arc melting. The amount of rare earth recovered in the alloys differed with the particular solute but in general, the elements with the smallest heat of sublimation were recovered least.

Zr undergoes a volume contraction when transforming from HCP to BCC. The alloys also exhibited this volume contraction with a decrease in the total contraction as the solute concentration increased. The temperature of the midpoint contraction on heating was assumed to be equivalent to the temperature of the allotropic phase boundary of the alloys. The allotropic phase boundary ap-

peared to be a function only of the atomic concentration of the rare earths studied. The transformation temperature increased up to the solid solubility limit approximately the same for all the elements investigated.. The solid solubility limits were related to the elastic strain energy due to the different atomic sizes. The similar elevation of the transformation is explained on the basis of similar electronic behavior of the rare earths. Other work on zirconium alloys has shown a relation between the superconducting transition and chemical valence of the solute. The chemical valence for the rare earths studied is usually +3. Electrical resistivity measurements on the Zr-rare earth alloys showed the resistivity varied similarly as a function of atomic concentration within the solid solubility region.

The research reported was done at Rensselaer Polytechnic Institute, and has been sponsored by the Atomic Energy Commission.

On the Habit Planes for Zirconium Hydride Precipitation in Alpha Zirconium Crystals:
E. S. Fisher and D. G. Westlake, Argonne National Laboratory.

The crystallographic orientations of zirconium hydride planes in a single crystal of alpha zirconium have been studied after various charging treatments in an attempt to explain a contradiction found in the literature. Langeron and Lehr reported, in 1956, that the plates occur along the (10·0) type planes, whereas Kunz and Bibb found them only on (10·2), (11·1) and (11·2) type planes. This study provides substantial evidence for the (10·0) planes and no evidence for the others. At least one other (10·1) type plane is sometimes observed. Possible explanations for the results reported by Kunz and Bibb will be presented. Further study is necessary to determine whether certain charging conditions can produce habit planes other than the (10·0) type planes.

Martensitic Transformations in Vanadium-Ruthenium Alloys:
James Perrin, Battelle Memorial Institute, and D. S. Lieberman, University of Illinois.

Transformations in two vanadium-ruthenium alloys of near equiatomic composition have been investigated. Hot stage microscopy, X-ray, resistivity, dilatometry, and preliminary magnetic susceptibility studies will be reported. These show a reversible phase transformation exhibiting no hysteresis which occurs over the temperature range from 175°K to 450°K, depending on composition.

This work was partially sponsored by the United States Atomic Energy Commission.

Field Emission Microscopy of Metal Crystal Nucleation:
K. L. Moazed, Ohio State University and G. M. Pound, Carnegie Institute of Technology.

Field emission microscopy was used to investigate the heterogeneous nucleation of metal crystals from the vapor on metallic substrates. Quantitative measurements were made for the nucleation of silver on tungsten. Critical supersaturations for nucleation of silver on tungsten field emitter tips at room temperature were measured and found to correspond to a Gibbs free energy change of approximately 2900 calories per cubic centimeter. The contact angle between solid silver and clean tungsten was computed from the data to be 84°. The mechanism of nucleation was determined to be (1) absorption in which the thermal beam flux of silver accumulated on the substrate (emitter tip) to a critical coverage of a few tenths of a monolayer, followed by (2) nucleation from the absorbed atoms to form silver crystallites. The field emission pattern clearly revealed the absorbed silver atoms and the stable, growing nuclei. This mechanism is different from that encountered in previous studies of nucleation from the vapor in that the desorption rate is so low that there is no metastable equilibrium with the incident beam of silver atoms. Accordingly, it was necessary to revise the existing theoretical treatment to accommodate this circumstance. Numerous qualitative observations were documented through the media of still and motion picture photography. Among these are:

(i) Surface diffusion of silver on tungsten occurs most rapidly on high index planes of tungsten.
(ii) The high index planes and the (III) planes of tungsten have the highest catalytic potency for silver crystal nucleation.
(iii) Growth and evaporation of the crystallites were observed, and the effects of capillarity were evident.

All-Institute Pre-Smoker Social Hour

6:00 pm — Chase-Park Plaza Hotel — Exhibit Hall

All-Institute Dinner-Smoker

8:00 pm — Chase-Park Plaza Hotel — Khorassan Room

TUESDAY, FEBRUARY 28, AM

ISD Physical Chemistry of Slags

9:00 am — Ambassador Hotel — Empire Room (Lobby Floor)

Chairmen:
J. F. Elliott, Massachusetts Institute of Technology
R. E. Boni, Armco Steel Corp.

TUESDAY, FEBRUARY 28, AM

Interfacial Tension Between Liquid Slag and Metal:
G. Worth and G. R. St. Pierre, Ohio State University.

Density of Liquid Iron Silicates:
J. Henderson, R. G. Hudson, R. G. Ward, and Gerhard Derge, Carnegie Institute of Technology.

Oxygen Potentials and Phase Equilibria in the Fe-Ca-O System:
E. T. Turkdogan, U.S. Steel Corp.

Determination of Blast Furnace Slag Basicity by a Fusion-Point Method:
K. H. Gee, Bethlehem Steel Corp.

Thermodynamics of Si-O:
F. D. Richardson and H. Ramstad, Royal School of Mines, England

EMD-MBD
Application of Carbon in the Pyrometallurgy Industry

(Sponsored by EMD Pyrometallurgy Committee and MBD Pyrolysis and Agglomeration Committee)

9:00 am — Ambassador Hotel — Coronation Room (Lobby Room)

Chairmen:
C. H. Aall, American Metal Climax, Inc.
T. L. Hurst, Monsanto Chemical Co.

Carbon and Graphite in the Metallurgical Industries:
N. J. Johnson, V. J. Nolan, and J. W. Shea, National Carbon Co., Division of Union Carbide Corp.

Use of Carbon in Industrial Furnace Linings:
W. B. Arnold, and L. H. Juel, Electrode Division, Great Lakes Carbon Corp.

Soderberg Anode Carbon in Cells for Electrolytic Production of Aluminum:
Ove Sandberg and Olav Bowitz, Electrokemisk AS, Norway.

Use of Permanent Graphite Molds for the Production of Precision Cast Steel Car Wheels:
H. H. Hursen, Griffin Wheel Co.

IMD Titanium Research

(Sponsored by IMD Titanium Committee)

9:00 am — Ambassador Hotel — Versailles Room (First Floor)

Chairman:
H. J. Middendorp, Wright Air Development Division.

An Examination of the Ti-Al-V System:
P. Farrar and H. Margolin, New York University.

Effects of Alpha-Soluble Additions (Aluminum, Carbon, Oxygen, Nitrogen) on the Structure and Properties of Titanium-Molybdenum Alloys:
F. C. Holden, H. R. Ogden and R. I. Jaffee, Battelle Memorial Institute.

This paper describes the metallurgical and mechanical characteristics of high-purity titanium-molybdenum alloys containing ternary and quaternary additions of aluminum, oxygen, and carbon. The

TUESDAY, FEBRUARY 28, AM

effects of these alloying additions on mechanical properties were determined for several microstructural conditions. The effects of alloying and heat treatment on the phase relationship and transformation kinetics of the several alloy system were determined.

The following ternary and quaternary alloys were studied: (1) nine Ti-Al-Mo alloys, containing up to 7 pct aluminum and 16 pct molybdenum; (2) two Ti-O-Mo alloys, containing 0.2 pct oxygen and up to 12 pct molybdenum; (3) two Ti-C-Mo alloys, containing 0.2 pct carbon and up to 12 pct molybdenum; (4) four Ti-O-Al-Mo alloys, containing 0.2 pct oxygen, up to 7 pct aluminum, and up to 12 pct molybdenum. The compositions were selected to correspond with those of binary Ti-Al and Ti-Mo alloys studied previously, and used as base-line properties.

The alloys were are melted, using electrolytic (high purity) titanium and alloying additions of comparable purity. Fabrication and heat treatments were conducted in a manner to minimize contamination. Essentially complete analyses for metallic and interstitial elements were made.

Heat treatments were designed to produce as wide a variety of microstructures as possible. In general, specimens were tested with (1) beta-quenched, (2) equiaxed alpha-beta, (3) transformed (acicular) alpha-beta, and (4) stabilized alpha-beta microstructures. Mechanical properties were evaluated by tensile, impact, and hardness tests. Transformation kinetics were studied using hardness measurements and metallography to follow transformations from the beta phase.

Results showed that aluminum additions slow the beta to omega transformation, whereas the carbon and oxygen additions increase the transformation rate. The effects of the alpha-solute addition thus include solid solution strengthening, and an altering of the transformation kinetics which may increase or decrease strength. The formation of strain-induced martensite is promoted by aluminum additions, such that yield strengths are reduced to very low values in certain alloys (specifically those at the 12 pct molybdenum level). Ductilities of alpha-beta alloys generally are increased by equiaxed microstructures, tensile strengths being about the same for equiaxed and transformed conditions. Many of these alloys are heat treatable to high-strength levels, as indicated by aging tests. Strength levels of the aged specimens normally are lowered by straining prior to aging.

Ti-Al-Cb Alloy Development:

C. R. Simcoe and J. McAndrew, Illinois Institute of Technology.

Titanium-base alloys containing additions of aluminum (5 to 17.5 pct) and columbium (15 to 30 pct) in various combinations exhibit properties which mark this class of alloy as unusual and potentially valuable. Experimental findings are presented which show that such alloys are forgeable, are oxidation resistant to 1000°C (1832°F), and have densities below 5 g/cm³ (0.181 lb/in.³). Some alloys had exceptionally high strength-to-density ratios in short-time tensile tests at temperatures up to 871°C (1600°F). Very limited stress-rupture testing showed one alloy to have a 40-hr life at 10,000 psi and 1700°F, or a 100-hr life at 13,000 psi and 1600°F, in air. A second alloy had a life of 97 hr at 10,000 psi and 1600°F, a 174-hr life at 25,000 psi and 1400°F, and over 1000-hr life at 10,000 psi and 1400°F. These were single-specimen tests.

Room-temperature ductility, stress-thermal stability, and heat treatment of this type of alloy are discussed in the light of preliminary experiments.

Crack Propagation Characteristics of Three Titanium Alloys: Ti-6Al-4V; Ti-4Al-3Mo-1V; and Ti-13V-11Cr-3Al:

A. J. Hatch, Titanium Metals Corp. of America.

A Reinvestigation of the Ti-Cr and Ti-V Systems:

F. Ermanis, P. Farrar, and H. Margolin, New York University.

Notch Sensitivity of Titanium Alloys:

H. R. Ogden, R. W. Douglass, F. C. Holden, and R. I. Jaffee, Battelle Memorial Institute.

The notch sensitivity of titanium alloys is affected by impurity content, microstructure, and heat treatment. Using notch tensile properties to evaluate notch sensitivity, three commercial titanium-base alloys were investigated over the temperature range of −320° to 300°F. The alloys studied were Ti-5Al-2.5Sn, Ti-6Al-4V, and Ti-2Fe-2Cr-2Mo. Six interstitial levels were studied for each alloy in four microstructural conditions. The alpha-beta alloys were studied in both the annealed and heat-treated conditions. All tests were conducted using ¼-inch diameter round tensile samples. The notched samples had a 60 per cent notch with a notch sharpness (a/r) of 53.

Notch sensitivity was increased by increased interstitial content, low temperature, and acicular-type microstructures. The heat-treated alpha-beta alloys were more notch sensitive than the same alloys in the annealed condition. In addition, sensitivity to slow-stain hydrogen embrittlement was observed at temperatures near 32°F at hydrogen levels well below the 150 ppm specification limit.

A Study of the Ti-Cu-Zr and Ti-Zr Systems and the Structure of Ti_2Cu:

Elmars Ence and H. Margolin, New York University.

The use of higher purity titanium and zirconium indicates a more sharply dropping alpha and beta transus than has been indicated in earlier work in composition below 45 wt. pct zirconium. The partial isothermal section of the Ti-Zr-Cu system at 750°C has been studied. The crystal structure of Ti_2Cu has been determined as tetragonal and when expressed as face-centered tetragonal, a = 4.16Å, c = 3.611Å, and c/a = 0.867.

Low Temperature Properties of Titanium Alloys:

D. L. Day, Titanium Metals Corp. of America.

IMD Dislocations

(Based on submission of research abstracts)

9:00 am Ambassador Hotel Marquis Room (First Floor)

Chairman:

W. W. Webb, Union Carbide Metals Co.

On the Yield Point in BCC Metals:

G. Schoeck, Comision Nacional de Energia Atomica (Argentina)

It has been observed that the yield stress in bcc metals depends on the grain diameter, d, according to the equation 1: $\sigma_Y = \sigma_i + kd^{-1/2}$. In a theoretical interpretation of this result by Petch and Cottrell the factor k is proportional to the stress to unlock a dislocation from an atmosphere of foreign atoms and should according to theory depend strongly on temperature. Further σ_i is interpreted as a frictional stress acting on dislocations in a pile-up supposedly caused by the Peierls stress and by the internal stress-field of precipitates.

More recent experiments have however shown that k is fairly independent of temperature at least between 120°K and 300°K and that the in this range temperature dependence of σ_Y is caused mainly by σ_i. Furthermore it has been observed that the temperature dependence of the flow stress (i.e. movement of free dislocations) is the same as that of the yield-stress.

Since all existing calculations on the temperature dependence of the locking stress do not take account properly of the position of the locking atoms it is feasible that unlocking of dislocations from atmospheres is practically independent of temperature. An interpretation of frictional mechanisms for σ_i can be given, but it can be shown that internal stressed due to precipitates would not increase σ_i as observed.

This leads to a different interpretation of the experimental data: σ_i can be interpreted as a back-stress acting at a Frank— Read source and the grain-size dependence becomes equation 2: $\sigma_Y^2 = \sigma_i^2 + k'd^{-1}$. The scattering of the experimental data is so large that they also fit equation 2.

On the Mechanism of Yielding and Flow in Iron:

Hans Conrad, Atomics International

The activation energy, activation volume, and frequency factor have been evaluated for yielding (delay time for yielding, upper yield stress, and lower yield stress) and flow (friction stress, flow stress and dislocation mobility) for various irons and steels from data in the literature. It was found that the value sof these flow parameters and their stress dependence were the same, within experimental error, for both yielding and flow and for all the materials considered. This suggests that either the same dislocation mechanism is controlling in every case or that one or more mechanisms possess approximately the same values for these parameters. The dislocation mechanism for which there was closest agreement between theoretical calculations and experimental data was overcoming the Peierls stress.

On the basis of the available experimental data and the present analysis it is suggested that the upper and lower yield stress in iron and steel represent the sudden generation of a large number of dislocations by the double cross-slip mechanism of Koehler and Orowan rather than the breaking away from a Cottrell atmosphere.

Strain Energy of Dislocation Networks:

J. C. M. Li, U.S. Steel Corp.

The strain energy of a crossed grid of screw dislocations is calculated using isotropic elasticity theory. It is found that a slight distortion from the stable structure requires a large amount of strain energy. In other words, recovery from the strained state can release a considerable fraction of the total stored energy without large discernible changes in dislocation substructure; this was experimentally observed by Bailey and Hirsch. For example, by simply rearranging into a stable structure (equivalent to a crossed grid of two sets of screw dislocations oriented at 90° with respect to each other) a crossed grid of two sets of parallel screw dislocations oriented at 90° with respect to each other can release the same amount of strain energy as is released by the annihilation of one set of parallel, like screw dislocations. This indicates that long-range stresses can exist in metastable dislocation networks without a long pileup of like dislocations. It also questions the measurement of dislocation density by stored energy alone. Possible rearrangements of dislocation networks in both fcc and bcc metals will be shown.

Long Range Pinning of Dislocations by Atomic Defects:
R. L. Fleischer, General Electric Co.

The pinning effect of atomic defects in the stress field of a dislocation is evaluated for defects positioned outside of the core region. The impurity distribution produced at an elevated temperature is assumed to be fixed at a lower temperature and the stress required to move the dislocation relative to its atmosphere is then calculated.

For such an atmosphere the stress is appreciable and varies slowly in such a manner that the solute distribution may be regarded as equivalent to a dislocation of fractional slip vector and opposite sign to the dislocation responsible for its existence. The presence of these long range restoring forces implies that unpinning of dislocations is appreciably more difficult than previous calculations would indicate.

When the strain field of a dislocation is saturated in the sense described by Thomson, the binding of a dislocation to its atmosphere is so tight that restoring stresses of the order of the theoretical strength extend many atomic distances from the dislocation. In this case stress-aided unpinning of dislocations is not to be expected.

Plastic Properties, Dislocation Velocities, and Charged Impurity Effects:
J. R. Patel and A. R. Chaudhuri, Raytheon Co.

The macroscopic plastic properties of dislocation-free silicon have been investigated over a temperature range 600°-1000°C. As has been noticed by other workers, a characteristic of the tensile deformation curves is the large drop in stress at the yield point. The yield phenomenon is not due to a dislocation-locking mechanism and contrary to previous findings, no strain-ageing has been observed. As has been shown for germanium, the macroscopic plastic properties can be explained on the basis of the dynamical behavior of dislocations in silicon. Dislocation velocities have been measured over a range of stresses and temperature. The temperature dependence of velocity has been found to be steeper than in germanium with an activation energy of 2.16 ev. Possible implications of the steeper temperature dependence will be discussed. Tensile deformation experiments have also been made on samples heavily doped with boron, gallium, and arsenic. Because of the acceptor-like behavior of vacancies in germanium, heavy doping with n- or p-type impurities modifies both the velocity of dislocations and plastic deformation characteristics. Contrary to our findings in germanium, there is no effect of n- or p-type impurities on the deformation of silicon. Since the charge of a vacancy in silicon is not known, it is possible either that the vacancies are not charged or, if charged, that the vacancy energy level lies very close to the center of the energy gap. Under either of these conditions no effect of heavy doping should be noticed.

Dislocation Velocities in Indium Antimonide:
A. R. Chaudhuri and J. R. Patel, Raytheon Co.

Conventionally defined "dislocation-free' crystals of indium antimonide have been grown by the Czochralski technique. Condensed vacancy loops are therefore present in the crystals and these markedly affect the motion of dislocations in the crystals, and are sources for the generation of dislocations at small stresses.

The macroscopic plastic properties of the crystals have been measured in tension under a constant rate of elongation. As is found in germanium and silicon, a marked yield drop is obtained, and this phenomenon is again ascribed to the numbers and velocities of dislocations taking part in the process of deformation. It turns out that, on a comparable T/T_m basis, indium antimonide has much the same macroscopic plastic properties as germanium.

Dislocation velocities have been measured by the method of Stein and Low, using both three point bending and compression as methods of stress application. The velocity data show first, indium antimonide, silicon and germanium have about the same stress dependence of velocity, second, on a comparable T/T_m basis, the magnitudes of the velocities in indium antimonide are about 30 times greater than in germanium, and third, the activation energy for dislocation velocity in indium antimonide is about 0.77 ev.

The inference is drawn from the higher velocities, that the number of mobile dislocations producing deformation in indium antimonide is several times smaller than is the number for germanium. Observations show that dislocation multiplication is not as extensive in indium antimonide as it is in germanium, thus supporting the inference regarding the lower number of mobile dislocations.

Dislocation Arrangement in Alpha Iron During Deformation and Recovery:
A. S. Keh, U.S. Steel Corp.

MBD-EMD

Chemical Processes

9:00 am Chase-Park Plaza Hotel Starlight Roof

Chairmen:
F. T. Davis, Colorado School of Mines.
S. P. Warren, Consulting Engineer.
Merrill McCarty, The Dow Chemical Co.

Scandium Recovery from Vitro Uranium Solutions:
L. D. Lash, Vitro Chemical Co. and J. R. Ross, U.S. Bureau of Mines.

A Mechanism Study of the Formation of Sodium Vanadate Compounds Under the Conditions of the Salt Roast Process:
W. H. Dresher, Union Carbide Nuclear Co.

A study has been made of the reaction mechanisms occurring during the salt-roast process to form water-soluble sodium vanadate compounds from the vanadium oxide compounds present in vanadium-bearing ores and concentrates. Two reactions have been identified, either of which lead to water-soluble compounds:

TUESDAY, FEBRUARY 28, AM

oxidation of sodium chloride by water vapor and oxidation of sodium chloride by oxygen. The former reaction has been found to be predominant in the salt-roast process.

A kinetic treatment of the data has shown that for the formation of sodium metavanadate, the rate-controlling step in the reaction in which sodium chloride is oxidized by water vapor is the decomposition of a species containing water and sodium chloride on the surface at the gas-solid interface. For the formation of sodium pyrovanadate, the rate-controlling step is the decomposition of a species containing water and sodium metavanadate on the surface. The transportation of water through the system, perhaps as a sodium chloride-water complex or a sodium metavanadate-water complex, and the actual chemical reaction are relatively fast compared to the initial surface decomposition step.

A similar treatment on the data for the oxidation of sodium chloride by oxygen has shown that the rate-controlling step is the actual transportation of oxygen from the gas-solid interface to the reactive site. This transportation is by a sequence of two diffusion processes. Transportation during the early stages of the reaction is by the diffusion of oxygen through a continuous layer of reaction product. After the first few minutes of reaction time a second diffusion process begins and is continuous for the remainder of the reaction. This second diffusion process involves the diffusion of oxygen through a layer of reaction product that has developed voids around which the oxygen must diffuse. The hindering nature of this latter process causes the rate of transportation of oxygen to the reaction site to be considerably slower than by the simple diffusion mechanism initially followed.

A thermodynamic treatment of the data has shown that the energy of activation for the decomposition of the water-sodium chloride species on the surface is -12.1 Kcal. per mole. For the decomposition of the water-sodium metavanadate species on the surface, the energy of activation is -15.1 Kcal. per mole.

The results of this study indicate the limiting mechanisms of the basic reactions occurring during the salt-roast process. These results suggest what variables should be examined in applying this information to actual plant operations.

Chemical Metallurgy of Tungsten:
K. C. Li, Wah Chang Corp.

The problems and limitations of milling processes are outlined. The chemical upgrading of tungsten ores and concentrates is described. The methods of extraction and preparation of pure tungstic oxide from ores and concentrates are discussed. A generalized flow sheet is given showing how impurities are removed in each step.

Technique of Gas Oxidation During Pulp Agitation:
N. M. Levine, Stein-Hall and Co., Inc., and W. M. Fassell, Aero-neutronics,

A laboratory study shows how the use of certain physical parameters derived with aid of the basic fundamental mixing equation enhanced the resolution of a conditioning problem involving gas oxidation.

In this study the problem of aerative conditioning to separate chalcopyrite and pyrite from cobaltite was simply effected with a sulphydrate collector and pH by proper choice of mixing variables. It was shown that for a given impeller type and geometry, selectivity is governed by a relation between the Froude Number and a "modified" Power Number multiplied by Impeller to Tank Diameter Ratio wherein the power factor is expressed as power intensity or horsepower per unit volume of contained pulp.

In applying this approach to other systems, other criteria would be used in place of selectivity.

ISD Howe Memorial Lecture

11:00 am Ambassador Hotel Empire Room

R. R. McNaughton and J. J. Golden, Chairmen

Speaker:
L. S. Darken, U.S. Steel Corp.

Subject:
Role of Chemistry in Metallurgical Research.

TUESDAY, FEBRUARY 28, PM

ISD Annual Luncheon and Business Meeting

12:15 pm Ambassador Hotel Caesar Room (Lobby Floor)

J. J. Golden, Chairman, Presiding

Speaker:
L. F. Gulley, Granite City Steel Co.

All-Institute Session

2:30 pm Chase-Park Plaza Hotel Khorrassan C

State of the Institute:
Joseph L. Gillson, President, AIME

Topic to Be Announced:
Hendrik Schuiling, Union Miniere du Haut-Katanga, Belgium.

TUESDAY, FEBRUARY 28

Met Soc — Social Hour

6:00 pm Ambassador Hotel Coronation Room

Met Soc — Annual Dinner

7:00 pm Ambassador Hotel Coronation Room

C. C. Long, President, *Presiding*

Presentation of Awards:

J. E. Johnson, Jr. Award to R. J. Wilson.
Champion H. Mathewson Gold Medal to K. T. Aust and J. W. Rutter
Robert W. Hunt Medal to Y. Doi and K. Kasai

Speaker:

R. J. Anderson, Battelle Memorial Institute

Subject:

Journey Into Ignorance.

WEDNESDAY, MARCH 1, AM

IMD Recovery and Recrystallization

(Based on submission of research abstracts)

9:00 am Ambassador Hotel Empire Room (Lobby Floor)

Chairman:

S. Weissmann, Rutgers University.

Recrystallization of Aluminum Single Crystals:

D. C. Larson and Bruce Chalmers, Harvard University.

The recrystallization behavior of single crystals of aluminum of 99.99 pct purity has been studied. The crystals were deformed in tension and the tensile axis orientations were <111>, <211>, <100>, and <110>. The crystals were sawed into specimens and electropolished to remove material deformed in sawing and to remove controlled amounts of material at the surface. Finally, the specimens were annealed at 640°C. and the incidence or absence of recrystallization observed.

It was observed that crystals with axes of tension along the <100> – <111> boundary of the stereographic triangle required approximately the same shear stress (resolved on the active slip systems) for recrystallization, namely 1.5 kg/mm². This shear stress corresponds to 13 pct resolved shear strain for the <111> orientations, 62 pct for the <211>orientations, and 130 pct for the <100> orientations. The <110> crystals behaved differently. Deformation bands were observed and a resolved shear stress of 1.1 kg/mm² (resolved shear strain of 16 pct) was necessary for recrystallization.

In addition it was observed that the spontaneously nucleated grains form more readily in the layers near the surface than near the interior of the deformed crystal. Consequently, the recrystallization can often be suppressed by removing a surface layer by electropolishing prior to annealing. For example if the <111> orientation is deformed to 0.5 kg/mm² it will not recrystallize. Between 0.5 and 1.5 kg/mm² increasing amounts of electropolishing are necessary to suppress recrystallization and at 1.5 kg/mm² no amount of electropolishing will suppress recrystallization.

Finally, it was observed that much less deformation is required to support growth than to induce nucleation.

This research was sponsored in part by The Office of Naval Research contract Nonr-1866 (27) and in part by the National Science Foundation.

Effect of Cold Working and Recrystallization on the Annealing Out of Vacancies in a Ag-Zn Alloy:

A. S. Nowick, International Business Machines Corp., and A. E. Roswell, Radio Corp. of America.

By observing the relaxation time for stress-induced ordering (the *Zener relaxation effect*) in a substitutional solid solution, it is possible to deduce the rate at which vacancies quenched into the alloy anneal out at relatively low temperatures. Previous work involved the effects of small amounts of deformation on the annealing out of vacancies in a Ag-33 atomic pct Zn solid solution. It was shown that the presence of dislocations enhances the rate of annealing out of vacancies. In the present work, heavily cold worked samples (deformed by drawing into wire) were quenched from 400°C, and the relaxation time was measured as a function of time at 70°C. It was not possible to observe a relaxation at 70°C until the sample had been recrystallized prior to quenching. However, even after recrystallization anneals at 500°C, vacancies were observed to anneal out at abnormally high rates. Only after an anneal at 600°C does the vacancy decay curve return to its normal level, characteristic of the fully annealed state.

These results suggest that, even after recrystallization, lattice imperfections which serve as condensation nuclei or as sinks for vacancies are present, and that the degree of perfection of the lattice continues to increase during grain growth.

This work was performed at Yale University where it was supported by the Office of Ordnance Research.

Texture Transition in High-Purity Silver and Its Correlation with Stacking Fault Frequency:

Hsun Hu, R. S. Cline, and S. R. Goodman, U.S. Steel Corp.

In a previous paper it was reported that the rolling texture of high purity silver was the common *silver type*, i.e. (110) [$\bar{1}$12], when the specimen was rolled at 0°C. A *copper type* rolling texture which can be described as approximately (124) [211] and (146) [21$\bar{1}$], was obtained when the specimen was rolled at 200°C. A detailed study of this texture transition as a function of rolling temperature was then followed.

The rolling textures determined for specimens rolled at three intermediate temperatures within the range 0° to 200°C showed a gradual transition from the *silver type* to the *copper type*. At 50°C, the texture was still of the *silver type*. Changes toward the *copper type* were clearly noted in the specimen rolled at 100°C. The texture of the specimen rolled at 150°C was essentially of the *copper type*, although the (100) [001] cube component (resulted from recrystallization during rolling) was considerably less than that in the specimen rolled at 200°C.

In connection with the idea that the deformation texture of a fcc metal may be dependent on its stacking fault energy, the stacking fault frequency of each specimen was determined from the peak shift measurements. The stacking fault frequency so determined for the specimen rolled at 0°C was approximately every 120 d_{111} spacings, and that for the specimens rolled at temperatures above 100°C could hardly be detected. A correlation of these data with the texture transition is presented, and the results are discussed.

Oriented Growth Mechanism of the Formation of Recrystallization Textures in Aluminum:

M. N. Parthasarathi and P. A. Beck, University of Illinois.

A high purity (99.997 pct) aluminum crystal of initial orientation (123) [4$\bar{1}$2] was rolled 80 pct, and artificially nucleated by rubbing one side with emery paper. It was found by quantitative pole figure methods that the thin recrystallized layer first formed at the rubbed side on annealing for 2½ min at 325°C was quite random, but that a sharp recrystallization texture developed on the other side of the sheet by means of selective growth of the random nuclei when the annealing was continued for 150 min at 325°C. All components of the recrystallization texture were related to the deformation texture by 40° rotations around [111] axes. The relative volume fractions of the various recrystallization texture components can be accounted for on the basis of differences in the mobility of boundaries of various orientations, with a fixed lattice orientation relationship between the recrystallized grains and the matrix.

The results obtained with an 80 pct rolled (110) [1$\bar{1}$2] crystal of an aluminum alloy containing Fe, Si and Zn in solution indicate that these solute contents do not impair the effectiveness of the oriented growth mechanism in producing a sharp recrystallization texture identical with that in a high purity aluminum crystal of the same orientation.

This work was performed under the sponsorship of the US Atomic Energy Commission.

A Study on the Texture Formation in Rolled and Annealed Crystals of Silicon-Iron:

Hsun Hu, U.S. Steel Corp.

Three single crystal strips of a 3 pct Si-Fe alloy with approximately (110) [001], (210) [001] and (100) [001] orientations were rolled at room temperature to 30 pct, 50 pct and 70 pct reductions in thickness. Their deformation and annealing textures were studied. In most cases, the change in texture upon recrystallization and subsequent growth can be described as rotations around the <110> axes. A [100] rotational relationship was also found to be prominent, particularly in the specimens given heavy reductions. In the 70 pct rolled (110) [001] crystal, the recrystallization texture can be either a (110) [001] or a (210) [001], depending upon the temperature of anneal. The behavior of the (210) [001] crystal is very similar in nature to that of the (110) [001] crystal, and the annealing texture of a 70 pct rolled specimen can be either a (210) [001] or a (410) [001]. The orientation of the original crystal may not be present as a very weak component in the deformation texture. In the present 70 pct rolled (100) [001] crystal, no such component was detected by either X-ray or metallographic examination; yet, a prominent component of the recrystallization texture is (100) [001]. These results indicate that the presence of a measurable minor component in the deformation texture is not a necessary condition for the formation of a recrystallization texture of that orientation.

A Mechanism for Recovery in Cold-Worked Body Centered Cubic Metals:

D. P. Gregory and G. H. Rowe, Pratt & Whitney Aircraft Div., United Aircraft Corp.

Low and Guard have presented evidence for a double cross slip deformation mechanism in single crystals of body centered cubic silicon iron. Deformation by this mechanism places screw dislocations of opposite sign on parallel slip planes separated by a number of lattice spacings. Work hardening appears to result from interaction of the stress fields of the unlike screw dislocations.

It is proposed that recovery of cold work during annealing occurs by a mechanism of annihilation of these unlike screw dislocations by thermally activated cross slip. The kinetics of the recovery mechanism are similar to those of the exhaustion theory of primary creep. Calculations with elastic theory of dislocations suggest the following characteristics for the recovery mechanism.

(1) Activation energy for initial recovery much less than that for self diffusion

(2) Increasing activation energy with increasing recovery, approaching the activation energy for self diffusion
(3) Logarithmic time dependence of recovery
(4) Shift to a lower spectrum of activation energies for increasing amounts of cold work
(5) Higher work hardening rates after recovery in specimens with increasing amounts of prior cold work
(6) Greater recovery in specimens which have been cold worked at lower temperatures
(7) More sluggish recovery in single crystals than in polycrystals

Recovery data for pure iron of Michalak and Paxton and data for columbium-1 pct zirconium alloy of Stewart, et.al., are presented in support of the proposed theory.

This work was performed in the CANEL Materials Laboratory of Pratt & Whitney Aircraft, Division of United Aircraft Corporation, under contract to the United States Atomic Energy Commission.

Mobility of <100> Tilt Grain Boundaries in High-Purity Lead:
J. W. Rutter and K. T. Aust, General Electric Co.

A new technique has been developed which permits control of the relative crystallographic orientations in high-purity bicrystals for grain boundary migration studies. The method is also applicable to the preparation, from high-purity materials, of accurately oriented, substructure-free single crystals or of bicrystals with a transverse grain boundary. This technique was used in the present investigation to prepare bicrystals of zone-refined lead for a study of the mobility of large-angle grain boundaries as a function of orientation difference, θ, about a common <100> tilt axis.

It was found that the temperature dependence of the grain boundary migration rate, in the range from 320 to 175°C, is a function of θ in the misorientation range studied from 15° to 45° about <100>. A plot of measured activation energy for boundary motion versus θ <100> tilt was obtained which shows the presence of cusps at orientation differences near 23°, 28°, and 37°. These three grain boundary angles, which gave the lowest activation energies, correspond to the predicted coincidence site orientation relationships about a common <100> axis.

These results clearly indicate that differences in structure must exist among the various large-angle grain boundaries. In addition, the data provide strong experimental evidence which demonstrates the value of the coincidence site model.

EMD Beryllium

(Sponsored by EMD Light Metals Committee)

9:00 am Ambassador Hotel Coronation Room (Lobby Floor)

Chairmen:
R. A. Lewis, Kaiser Aluminum and Chemical Corp.
C. W. Schwenzfeier, The Brush Beryllium Co.

Outlook for Beryllium Ore Resources:
Norman Williams, Beryllium Resources Inc.

Extraction of Beryllium by the Use of Fluorides:
B. Kopelman, The Beryllium Corp.

Research on the Purification of Beryllium:
J. P. Pemsler, S. H. Gelles, and A. R. Kaufmann, Nuclear Metals, Inc.

Powder Metallurgical Processing of Beryllium:
K. G. Wikle, The Brush Beryllium Co.

Beryllium as a Structural Material for Aircraft, Missiles, and Spacecraft:
L. A. Riedinger, Missiles and Space Division, Lockheed Aircraft Corp.

EMD Hydrometallurgical Treatment of the More Common Metals

(Sponsored by EMD Hydrometallurgy Committee)

9:00 am Ambassador Hotel Embassy Room (Lower Level)

Chairmen:
F. Stephens, Jr., Battelle Memorial Institute.
W. C. Hazen, Kerr-McGee Oil Industries.

Treatment of Complex Sulfide Ores of Copper, Lead, and Zinc—Study in Metallurgical Research and Development—Part I, Metallurgical Research:
J. E. Hanway, Jr., and G. R. Smithson, Jr., Battelle Memorial Institute.

Treatment of Complex Sulfide Ores of Copper, Lead, and Zinc—Study in Metallurgical Research and Development—Part II, Pilot Plant Development:
R. E. Lund, D. E. Warnes, and J. A. Morgan, St. Joseph Lead Co.

Hydrometallurgical Treatment of Mercury Ores:
W. A. Stickney and J. W. Town, U.S. Bureau of Mines

Application of Continuous Centrifuges to Hydrometallurgical Processes:
W. F. White, Bird Machine Co.

ISD Thermodynamics of Liquid Iron

9:00 am Ambassador Hotel Versailles Room (First Floor)

Chairmen:
W. M. McKewan, U.S. Steel Corp.
D. V. Ragone, University of Michigan

C-O Equilibrium in Liquid Iron:
Tasuku Fuwa and John Chipman, Massachusetts Institute of Technology.

Activity of Oxygen in Liquid Fe-P:
Daniel Dutilloy and John Chipman, Massachusetts Institute of Technology.

Thermodynamics of Liquid Fe-C-Si Alloys at Temperatures Above 1200°C:
W. C. Phelps, Jr., and R. Schuhmann, Jr., Purdue University

Thermodynamic Properties of Oxygen in Binary Alloys of Iron, Cobalt, and Nickel:
E. S. Tankins and N. A. Gokcen, University of Pennsylvania

Sulphur Equilibria Between Gases and Calcium Ferrite Melts:
E. T. Turkdogan and L. S. Darken, U.S. Steel Corp

IMD Diffusion and Surface Phenomena

(Based on submission of research abstracts)

9:00 am Ambassador Hotel Marquis Room (First Floor)

Chairman:
W. W. Mullins, Carnegie Institute of Technology.

Relative Energy and Etching of Coherent Twin Boundaries in Germanium:
R. S. Wagner, Bell Telephone Laboratories, Inc.

The relative energy of coherent twin boundaries in germanium has been measured by the dihedral angle method. It was found that the energy of a coherent twin boundary is about 3 to 4 pct of the energy of a high angle <211> tilt boundary. The etching behavior of twinned germanium crystals was investigated on surfaces perpendicular to the twin boundary. The etching of a coherent twin boundary depends markedly on surface orientation. Using a Superoxol etch on the {211} faces perpendicular to the twin boundary, a striking anomaly in etching was found. On face A (the face from which the {111} planes which are parallel to the trace of the twin plane are seen as a ridge structure), etching leads to the formation of a well defined ridge where the twin boundary intersects the {111} surface. On the opposite face (face B, from which the {111} planes which are parallel to the trace of the twin plane are seen as a groove structure), etching leads to the formation of a faint groove at the twin boundary. The formation of an etch-ridge on face A at the twin boundary can be reproduced by employing different etching reagents. A mechanism is proposed for the peculiar etching behavior of coherent twin boundaries in germanium.

Low Angle Grain Boundary Self-Diffusion in Lead:
H. von E. Doering and W. R. Upthegrove, University of Oklahoma.

Controlled orientation symmetrical bicrystals are grown utilizing a modified Chalmer's technique. Both lattice and grain boundary self-diffusion are obtained by plating Pb^{210} isotope on a specimen surface perpendicular to the grain boundary and subjecting this couple to appropriate diffusion anneals. Following the diffusion anneal, the specimen is sectioned along a plane mutually perpendicular to the boundary and the diffusion interface and then metallographically prepared. Premeable base autoradiographic stripping film is applied to this surface and exposed. The film, developed in situ, reveals both lattice and grain boundary penetration of Pb^{210}. Standards are established to relate blackening of film to the isotope concentration and are utilized to determine the depth at which a given concentration occurs. The equations of Fisher, Whipple, and Borisov and Golikov are used to analyze these data.

Previous investigators have disagreed concerning the orientation

dependence of ΔH_b, the activation energy (as per Arrhenius equation), for grain boundary self-diffusion. The observed discrepancies were greatest at values of low misfit. Few data are presently available for θ or ϕ less than 15°. In this research the self-diffusion of lead in low angle symmetrical tilt and twist boundaries is investigated.

The research was sponsored by the Metallurgy Branch of the Office of Naval Research.

A Model for the Calculation of Grain Boundary Diffusion Concentration Curves:

H. von E. Doering, W. R. Upthegrove, G. W. Denison, and H. A. Walls, University of Oklahoma.

The current model of grain boundary diffusion, proposed by Fisher, assumes a narrow semi-infinite slab of high diffusivity material embedded between two semi-infinite slabs of low diffusivity material. Solutions for this model have been reported by Fisher, Whipple, and Borisov and Golikov. These solutions have been successfully employed to analyze most reported grain boundary diffusion data, but tend to show unusually large deviations under conditions where D_{GB} is not several orders of magnitude greater than D_L.

The existing model assumes a discontinuous diffusion coefficient, characterized everywhere inside of the fixed-width boundary slab as D_{GB} (constant) and everywhere outside of the slap as D_L. A modified model which substitutes a $D(x)_{effective}$, a continuous function, symmetrical about the boundary and dependent upon the distance normal thereto.

Let $D(x)_{eff} = D_L + D'_{GB}\, g(bx)$

where D_L is the conventional lattice self-diffusion coefficient, D'_{GB} is the maximum value of $D(x)_{eff}$ in excess of D_L, x is the distance normal to the free surface, b is a constant with units of reciprocal length, and g (bx) is an arbitrary, continuous, symmetrical (in x) function.

The differential equation of diffusion for this system becomes:

$$\frac{\partial C}{\partial t} = D(x)\,\nabla^2 C + \frac{\partial D(x)}{\partial x}\,\frac{\partial C}{\partial x}$$

Defining the following variables, constants, and dimensionless groups:

D_L, C, C_o, t have their normal meaning
D'_{GB}, $D(x)_{eff}$, b, x as defined above
y = distance normal to the initial diffusion interface

$$\psi = \frac{C}{C_o}$$

$$\alpha = bx$$

$$\beta = by$$

$$\theta = \frac{b^2 D_L t}{D'_{GB}}$$

$$\kappa = \frac{D'_{GB}}{D_L}$$

the equation becomes: $\dfrac{\partial \psi}{\partial \theta} = [1 + \kappa\, g(\alpha)]\,\nabla^2\psi + \kappa\,\dfrac{\partial g(\alpha)}{\partial \alpha}\,\dfrac{\partial \psi}{\partial \alpha}$

The function $g(bx) = e^{-(bx)}$ is studied. Available data are evaluated utilizing the proposed model.

This research is sponsored by the Metallurgy Branch of the office of Naval Research.

A Treatment of Diffusion Path Networks in Crystal Lattices:

R. H. Condit, University of California.

In any but the simplest crystal structures an understanding of the diffusion mechanism must include a treatment of the full network of paths which an atom may take in going from one normal lattice site to another. Such paths may be branched and will likely include stopping points at metastable sites along the way. An atom may therefore have several routes available to it for diffusion and if intrinsic disorder is large it may reside in the metastable sites for significant periods of time. Under such circumstances the usual log D versus 1/T plot may not be linear but may have a slope at low temperatures either greater or less than the high temperature slope. An illustrative analysis of the networks for cation diffusion in the nickel arsenide and wurtzite structures is carried out under the assumption that the motions of the cations in the two cases may be regarded as confined to jumps between interstices within the hexagonal close packed lattices of the immobile anions.

This work was performed under auspices of the U.S. Atomic Energy Commission.

Determination of Torque Terms on Copper Surface:

W. M. Robertson and P. G. Shewmon, Carnegie Institute of Technology.

Along the lines where twin boundaries intersect external surfaces, grooves or humps form due to the equilibrium between the various surface tensions and the torque terms tending to rotate the external surfaces into low energy configurations. By measuring the diheral angles at the intersections of pairs of twin boundaries with free surfaces, torque terms have been determined for OFHC copper. The polycrystalline samples were annealed near the melting point of copper in dry hydrogen. After photographing the grooves, the orientations of the crystals were determined using an optical goniometer.

The torque terms were determined on surfaces of several different crystallographic orientations. Surface near the low index planes (100), (110), and (111) tend to rotate toward the low index plane. The torque terms on surfaces near (100) and (111) planes were found to be quite appreciable, being at least as large as the twin boundary energy. The torque terms on surfaces near (110) were found to be smaller than on surfaces near the other two low index planes.

The torque term was found to vary as a function of orientation. By plotting the torque term versus the angle from a low index plane, a semi-quantitative picture of the variation of surface energy with orientation was obtained.

This work was supported by the Atomic Energy Commission and the National Science Foundation.

Determination of the Surface Migration Activation Energy and the Surface Tension of Molybdenum in the Solid State:

L. C. Crouser and R. W. Strayer, Linfield Research Institute.

The surface tension and surface migration constants of metals in the solid phase can be measured by the use of pulsed field emission microscopy, through an application of C. Herring's theory of transport phenomena in solids to field emission cathodes. (1) This paper reports the measurement of the activation energy of the surface migration of molybdenum atoms over a molybdenum substrate, and the surface tension of molybdenum in the region of a (110) surface at a temperature of 1700°K. These measurements were made on very pure metals at high vacuum and high temperature which assured surface cleanliness of the metal.

The procedure for the measurement of activation energy consists of determining the rate of removal of atom layers from the tip of a heated molybdenum field emission cathode as a function of temperature. (2) The removal of each atom layer is identified by the shrinkage of a ring of emission visible on a low index plane in the emission pattern of the cathode. The emission pattern is made visible by the application of one microsecond voltage pulse, at a repetition rate low enough so that the time average of the induced electrostatic force has a negligible effect upon the surface migration rate. The activation energy is then determined from the Boltzmann relationship for diffusion, $D = D_o e^{-Q/RT}$.

The surface tension is determined by finding the value of the electric field necessary to prevent the removal of atom layers from the tip of the field emission cathode held at a given temperature. By equating the electrostatic stress due to the surface field to the stress due to surface tension, a value for the surface tension is given by $\gamma = r\,\dfrac{F_o^2}{8\pi}$, where r is the radius of the cathode and F_o is the electric field required to stop surface migration in the (110) region of the cathode.

The average value of the surface activation energy of molybdenum atoms on molybdenum and the surface tension of molybdenum in the solid state at 1700°K was found to be the following:

$$Q = 2.28 \pm .06 \text{ ev/atom or } 64 \pm 1.4 \ \frac{\text{kilocalories}}{\text{mole}},$$

$$\gamma = 2200 \pm 200 \ \frac{\text{dynes}}{\text{cm}}.$$

This work was supported by the U.S. Navy, Office of Naval Research.

MBD-EMD-ISD
Basic Science Symposium on Kinetics and Design (I)

(Sponsored by Basic Science Committee of Minerals Beneficiation Division)

9:00 am Chase-Park Plaza Hotel Starlight Roof

Chairmen:
P. L. deBruyn, Massachusetts Institute of Technology
R. J. Charles, General Electric Co.
T. B. King, Massachusetts Institute of Technology

Reaction Kinetics in Metallurgy:

Henry Eyring, University of Utah.

Because of the complicated chain of reactions which are important in such processes as corrosion or combustion many people despair of reducing the scientific studies to useful practice. Such pessimism is not justifiable. Recently we were able to prove, building on the work of Ramachandran and King, that iron smelting is an electrochemical process in which oxygen ions from the slag combine with carbon in the iron to make CO gas and leave electrons on the molten metal which combine with either sulfur which passes into the slag or with some metal atom which passes from the slag to the metal.

We have studied the transients and steady state potentials of various metal electrodes in electrolyte solutions as effected by composition of the metal, composition of the electrolyte, composition of gases present, strain of the electrode and diffusional effects induced by stirring. This is interesting because of the light it throws on corrosion and the various catalytic processes occurring at the electrodes. The phenomena are discussed in terms of absolute reaction rate theory.

The theory of significant structures of liquids has enabled us to calculate the thermodynamic and transport properties of various liquids including surface tension. The detailed picture developed leads us to a better understanding of the abruptness of the transition between phases when only short range forces are involved. A brief summary of other interesting results and possibilities will be given.

Mass Transfer:

T. B. King, Massachusetts Institute of Technology.

The importance of mass transfer in the kinetics of metallurgical reactions is examined. For solid system most mass transfer problems can be described by fairly exact mathematical representations. Where fluids are involved, however, the mathematical description of mass transfer becomes much more complex. Theories of mass transfer in fluids, especially at fluid-solid boundaries, are reviewed

and examples are given where experimental information is available. It is concluded that a generalized theory is unlikely to appear, and that particular cases are best dealt with by the principles of similitude.

Theory and Application of Model Studies:
Kun Li, Jones & Laughlin Steel Corp.

Theory of models is briefly reviewed in general terms. Three examples, ingot solidification, mixing of solids and flotation conditioning are given to illustrate the application of model technique. In all three cases the possibility of model approach which could be advantageously taken is not known to have been explored.

Liquid Extraction Principles and Application to Nuclear Fuel Processing:
David Miller, International Institute of Nuclear Science and Engineering, Argonne National Laboratory.

The principles of solvent extraction are well known and if the equilibrium behavior are available, it is possible by simple experiments to provide the rate data necessary to design equipment.

Basic solvent extraction principles are discussed and illustrated by a new process of importance to metallurgical engineers. The use of liquid metals as extractions for metals in the solid and liquid state has attracted attention as economical means of reprocessing nuclear fuels. Such processes can retain the fuel in the metallic state avoiding the expensive dissolution and reduction steps associated with aqueous processing or fluoride volatility processes.

Extraction of plutonium by magnesium from uranium blankets in breeder reactors and other extraction processes are illustrated by flow sheets. Problems of materials of construction and equipment design are pointed out.

AIME Engineering Management

9:00 am Chase-Park-Plaza Hotels Tiara Room

(Program to Be Announced)

IMD Annual Business Meeting

10:45 am Ambassador Hotel Empire Room

T. A. Read, Chairman, Presiding

IMD Institute of Metals Lecture

11:00 am Ambassador Hotel Empire Room

C. C. Long and T. A. Read, Presiding

Speaker:
David Turnbull, General Electric Co., Schenectady, New York

Subject:
The Liquid State and the Liquid-Solid Transition

WEDNESDAY, MARCH 1, PM

EMD Annual Stag Luncheon and Business Meeting

12:15 pm Ambassador Hotel Caesar Room (Lobby Floor)

Reinhardt Schuhmann, Jr., Chairman, Presiding

Presentation of EMD Award to S. K. Basu and H. H. Kellogg.

Speaker: A. C. Weber, Laclede Steel Co.

Subject: *Where Is Our Strength?*

IMD Direct Observation of Imperfections in Crystals I: Review Lectures

(Sponsored by IMD Chemistry and Physics of Metals Committee)

2:00 pm Ambassador Hotel Empire Room (Lobby Floor)

WEDNESDAY, MARCH 1, PM

Chairmen:
J. B. Newkirk, Cornell University.
J. H. Wernick, Bell Telephone Laboratories.

Electron and Optical Microscopy:
J. W. Mitchell, University of Virginia.

Topographical Diffraction Methods:
W. W. Webb, Union Carbide Metals Co.

Field Emission Microscopy:
Erwin Muller, Pennsylvania State University.

EMD Hydrometallurgical and Other Treatment of the Refractory and the Rarer Metals

(Sponsored by EMD Hydrometallurgy Committee)

2:00 pm Ambassador Hotel Coronation Room (Lobby Floor)

Chairmen:
K. C. Vincent, Food Machinery and Chemical Corp.
W. L. Howes, Metallurgical Consultant

Hydrometallurgy of Tungsten and Vanadium:
Blair Burwell, Minerals Engineering Co.

Cesium and Rubidium:
C. E. Berthold, American Potash and Chemical Corp.

Thorium-Magnesium Alloy by Molten Salt Extraction:
F. W. Drosten, Vitro Chemical Co.

Properties and Handling Procedures for Rubidium and Cesium Metals:
T. D. Brotherton, O. N. Cole, and R. E. Davis, American Potash and Chemical Corp.

IMD Deformation and Strengthening

(Based on submission of research abstracts)

2:00 pm Ambassador Hotel Embassy Room (Lower Level)

Chairman:
M. R. Achter, Naval Research Laboratory

Deformation of Molybdenum-Rhenium Single Crystals:
Alan Lawley and R. Maddin, University of Pennsylvania.

High purity single crystals of binary Mo-Re alloys containing 5.4, 18, and 35 atomic pct Re were grown from arc cast or sintered bar using the electron bombardment floating zone technique. Tensile tests were made at −196°C, 25°C, and 200°C using strain rates in the range 4×10^{-5} to 1×10^{-3} in./in./sec. For the Mo-5 Re and Mo-18 Re alloys, deformation is by slip only, on {110} and {112} planes with a <111> slip direction. In the case of the Mo-35 Re alloy, deformation proceeds by twinning and slip Twinning is on {112} planes with slip on {110} and {112}. If it is assumed that the stress needed for slip on {110} is equal to that for slip on {112}, then it is found that the slip elements operative are consistent with the maximum resolved shear stress criterion.

From the data, the critical resolved shear stress is calculated as a function of rhenium content. The marked effect of 35 atomic pct Re in increasing ductility and U.T.S. is discussed in relation to the stress-strain curves and other pertinent data.

This research was sponsored by the Office of Naval Research.

Deformation Modes of UO_2 Single Crystals:
E. J. Rapperport and A. M. Huntress, Nuclear Metals, Inc.

Single crystals of uranium dioxide were deformed in compression at temperatures in the range 700°C to 1900°C. The most active slip plane at all temperatures was {100}, with {110} and {111} becoming more active as the temperature was increased. Fracture on {111} was observed with a single instance of {110} fracture recorded.

Estimates of the relative magnitudes of the critical resolved shear stresses were calculated by bracketing stress values for active and dormant slip planes.

This work was sponsored by the U. S. Atomic Energy Commission.

WEDNESDAY, MARCH 1, PM

Dislocation Structure of Bent Zinc Crystals:

P. P. Sinha and P. A. Beck, University of Illinois.

High purity zinc crystals were grown from the melt by the soft mold technique, and bent in such a way that the {0110} was parallel to the bending axis and the [2110] slip direction enclosed approximately 45° with the bending radius. A dislocation etching technique was developed, which reveals the edge dislocations introduced on bending, without the necessity of decoration. It was found that crystals bent either at room temperature or at liquid nitrogen temperature had approximately half of the edge dislocations arranged in low angle tilt boundary segments. The fact that no observable change occurred in the dislocation structure on annealing up to 225°C for 1,000 minutes suggests that the observed low angle boundaries were formed directly on bending, rather than by a thermally activated polygonization process, which might have been thought to take place at room temperature immediately after bending.

This work was performed under the sponsorship of the US Atomic Energy Commission.

Observations on the Ductility and Fracture of Recrystallized Chromium:

R. E. Hook, A. M. Adair, and H. A. Lipsitt, Aeronautical Research Laboratory, Wright Patterson Air Force Base.

Chromium wires prepared by the Bureau of Mines were recrystallized by resistance heating in an argon atmosphere and then deformed by bending as a cantilever at room temperature. The as received 0.040 in. diam wires were in a heavily cold worked condition and were ductile in bending at room temperature. However, these wires were quite brittle after a recrystallization anneal in vacuum at 1150°C. A 0.050 in. diam wire, being less severely worked, was neither ductile before or after a recrystallization anneal. Typical interstitial impurity contents are: 200 ppm oxygen, 8 ppm hydrogen, and 50 ppm nitrogen.

After recrystallization by resistance heating, a few single crystals were formed in the center of the wires and a grain size gradient existed from the center to the ends. The single crystals and adjacent large grain size area could be bent ninety degrees at room temperature with an accompanying tensile strain in the outer fibers of about fifty percent. The smaller grain size portions of the rod exhibited very little room temperature ductility and cleavage fractures were observed.

Many of the fracture surfaces examined consisted of both intergranular and transgranular fractures. This is believed to be the first reported evidence for even a small amount of intergranular fracture in chromium. In many specimens, is appeared that fracture initiated at a grain boundary and then propagated through the grains to failure. An interesting feature associated with the intergranular fracture is the appearance of an idiomorphic phase in the grain boundaries. The appearance of this phase suggests a segregation of impurities at grain boundaries which results in the initiation of fracture, somewhat analogous to the observations made on molybdenum and tungsten.

Acoustic Emission Under Applied Stress:

B. H. Schofield, Lessells and Associates, Inc., and Bruce Chalmers, Harvard University.

A brief description of the phenomenon of acoustic emission of metals is presented along with the techniques used for its detection and recording. Investigations show there is an intimate relationship of the emission to the deformation behavior of the materials and that it may provide additional insight into the specific activity of deformation mechanisms. Tests on both zinc and aluminum single crystals of various crystallographic orientation show the similarity of the emission characteristics to the specific strain and strain rate behavior of the specimen. Quantitative values obtained in the studies describe the physical relationship of the number of acoustic pulses which have occurred; i.e., total accumulated acoustic pulse density, to the observed strain. The data show displacements in the order of 10^{-8} cm per acoustic signal, values in the realm of micro-deformation mechanics. Tentative physical interpretation of the emission sources is suggested.

Structure of Shock-Loaded Single Crystals of Copper:

R. J. DeAngelis and J. B. Cohen, Northwestern University

C. S. Smith recently reported a metallographic study of shock loaded copper single crystals which were oriented with the [111], [110], [112], and [100] directions parallel to the direction of a plane shock wave of 300 kb. The [100] crystals did not show deformation markings (present in the other crystals) which resemble mechanical twins. It has been found in this investigation that at a pressure of 435Kb a crystal with [100] parallel to the shock direction displays the same markings as the other orientations.

Using Smith's crystals, a study of X-ray line shape for particle size and strain has been made with the Fourier methods developed by Warren and Averbach. All crystals were examined in the [110] direction parallel to the shock front. The strain distribution in the crystals with the [111] and [110] directions parallel to the shock direction were Gaussian and the rms strain decreased rapidly from 0.0012-0.0014 over 30A to 0.0008-0.0009 over 130A, whereas in the crystal with the [100] direction parallel to the shock direction the rms strain was constant at 0.0012 over the same distance, and the strain distribution was quite different.

The results of the Fourier analysis of diffraction peaks taken from copper single crystals shock-loaded at different pressures and examined in several crystallographic directions will also be presented.

The research was supported in part by ARPA.

Hard Particle Strengthening of Refractory Metals Through Internal Oxidation:

A. S. Bufferd and Klaus M. Zwilsky, New England Materials Laboratory, Inc., and N. J. Grant, Massachusetts Institute of Technology.

Molybdenum-0.5 pct titanium, molybdenum-1.5 pct titanium, and molybdenum-0.5 pct columbium were hard particle strengthened by internal oxidation. Superior properties, as measured by stress-rupture tests at 1800°F in argon, were obtained with the internally oxidized material. The 100-hr rupture life of Mo-0.5 pct titanium was increased from 50,000 psi to 60,000 psi by internal oxidation. For the Mo-1.5 pct Ti composition, the 100-hr rupture life was 54,000 psi for the internally oxidized material as compared to 22,000 psi for the as received powder metallurgical product. The ductility values of the internally oxidized material obtained in stress rupture, were quite promising when considering the increase in strength properties. Reduction of area values were 30 to 40 pct and 12 to 18 pct for elongation. These values are comparable to the arc cast material. Internal oxidation was achieved through the formation of an oxide film on chips prepared by machining bar stock. Oxygen diffusion to the interior of the chips at high temperatures resulted in the formation of fine titania particles. Electron microscopy was used in evaluating the resulting structures. The titania dispersion was present as needles 0.4μ in length and 0.1μ in width. In the case of the columbium alloy, the resulting dispersion was Cb_2O_5, .07 to .1μ in diameter.

This program was sponsored by the U.S. Navy Bureau of Aeronautics.

Effect of Stress on Grain Boundary Sliding in Creep:

N. R. Adsit and J. O. Brittain, Northwestern University.

Bicrystalline specimens of high purity zinc were tested in creep so that the state of stress in the boundary was pure shear. Tests were carried out at 200°C in a vacuum of better than 5 x 10^{-4} mm of Hg and at 350°C in argon at a pressure of approx ½ atm.

Visual observations of the specimens were made during the course of the testing by observing the movement of fiduciary marks. From the latter it is concluded that grain boundary sliding (GBS) was cyclic and that there was no regular periodicity. The data also showed that different positions along the grain boundary on the same specimen did not have the same periodicity. No grain boundary cracks were detected.

Measurements made after the completion of the test showed that the maximum grain boundary sliding was proportional to the resolved shear stress on the slip plane rather than the boundary orientation. These and other data suggest that the deformation process is the controlling factor in grain boundary sliding.

A model is proposed whereby boundary ledges which are the obstacles to GBS are smoothed off by the influx of vacancies from the deformation process thus facilitating GBS.

This research was supported by the U.S. Air Force through the Air Force Office of Scientific Research of the Air Research and Development Command.

Grain Boundary Shear in the Vicinity of Triple Points During Creep:

M. E. Knibbeler, A. W. Mullendore, and N. J. Grant, Massachusetts Institute of Technology.

Grain boundary sliding in the vicinity of triple points was studied in three and four grained specimens of aluminum, aluminum-1 percent magnesium, and aluminum-2 percent magnesium, subjected to constant strain rate (2 percent per hour) creep at 515° with elongations from 1 to 12 percent. The triple points were removed by drilling very small (0.005 to 0.013 inch diameter) holes through the specimen prior to testing. After testing, grain boundary displacements were determined as a function of distance along the boundary from the edge of the hole by the offset of reference lines on the surface. Thus, an indirect evaluation of the effect on grain boundary sliding of highly localized stress concentrations at the triple point was obtained. In particular, the presence or influence of a high hydrostatic stress, proposed by Zener, is assayed. The results obtained show little change in sliding due to the absence of the triple point. The magnitude of grain boundary displacements and their variation as a function of distance from the triple point is not noticeably changed. Restraint to grain boundary sliding in polycrystals is thus not attributable to highly localized stress concentrations at the triple points.

Grain Boundary Sliding During Creep of an Aluminum -2 Percent Magnesium Alloy:

A. W. Mullendore and N. J. Grant, Massachusetts Institute of Technology.

Grain boundary sliding was studied in polycrystal and bicrystal specimens of aluminum-2 percent magnesium at 500° and 700°F, the bicrystal specimens having random grain and grain boundary orientations. The specimens were deformed in tension at a constant strain rate of 2 percent extension per hour. Sliding was determined by the displacement of surface reference lines, when possible, on two perpendicular surfaces to obtain the sliding vector in space. The sliding was studied as a function of total elongation and orientation factors.

The observed amount and direction of sliding was found to depend on the relative orientations of the grains forming the boundary, the active slip planes, and the orientation of the boundary. It is concluded that grain boundary sliding is to a large extent induced by slip crossing the grain boundary, the sliding representing the unresolved component of shear when this takes place. The contribution of sliding which is independent of this action is of comparable magnitude only at low elongations.

Role of Very High-Strain Rates and Temperatures on the Deformation and Fracture Characteristics of Fe-S, Fe-S-O, and Fe-S-Mn Alloys:

S. Ogawa, T. B. King, and N. J. Grant, Massachusetts Institute of Technology.

Utilizing strain rates approaching 100 pct per second at temperatures from 1600° to 2400°F, the effect of sulfur additions up to

0.1 pct was studied on the ductility and fracture characteristics of pure iron in cast form. Additions of oxygen and manganese were made to the Fe-S melts to determine the effects of these elements over the same temperature and strain rate ranges.

Brittle fractures, independent of strain rate, resulted from the presence of the liquid FeS phase. Ductility was restored either when the liquid FeS dissolved in austenitic iron at the higher temperatures or when this phase had its melting point raised and its mode of distribution changed to the spheroidized form by both oxygen and manganese additions.

Homogenizing treatments led to mixed responses, depending on the sulfur content and the temperature of the test.

The relationships among the variables composition, temperature, strain rate and ductility are delineated in these three alloy systems.

ISD Application of Thermodynamics

2:00 pm Ambassador Hotel Versailles Room (First Floor)

Chairmen:
P. J. Koros, Jones & Laughlin Steel Corp.
T. P. Floridis, U.S. Steel Corp.

Equilibrium in the Fe-Al-O System:
R. A. Walsh and S. Ramachandran, Allegheny-Ludlum Steel Corp.

Thermodynamics of the Blast Furnace Process:
Valentin Koump, U.S. Steel Corp.

A Thermodynamic Model for the Deoxidation of Alloy Steels:
R. A. Walsh, S. Ramachandran, and J. C. Fulton, Allegheny-Ludlum Steel Corp.

Activities of Oxides in CaO-FeO-Fe_2O_3 Melts:
E. T. Turkdogan, U.S. Steel Corp.

Considerations in the Kinetics of Phosphorus and Sulfur Removal From Basic Electric Steels:
John Zotos, Northeastern University, and Eugene DeLuca, Watertown Arsenal.

IMD
Deformation of Semiconductors

(Sponsored by IMD Semiconductors Committee)

2:00 pm Ambassador Hotel Marquis Room (First Floor)

Chairman:
F. L. Vogel, Radio Corp. of America

Dislocation Velocities and the Role of Electrically Active Impurities in the Deformation of Dislocation-Free Semiconductor Elements and Compounds:
J. R. Patel and A. R. Chaudhuri, Raytheon Co.

"Dislocation-free" crystals of both the semiconductor elements and compounds, prepared by the Czochralski technique, deform at quite low stresses. It is postulated that such low stresses are due to the presence of condensed prismatic loops incorporated during growth.

The deformation characteristics are explained on the basis of the dynamical behavior of dislocations, i.e., on the numbers and velocities of mobile dislocations generated during the deformation. Direct measurements of the dislocation velocities have been made for Si, Ge, Ga-Sb and In-Sb crystals. The velocities are not the same at comparable homologous temperatures, and the fraction of dislocations that are mobile, under comparable conditions of deformation, is not the same for all the different types of crystals. The temperature dependence of the dislocation velocities shows a single activation energy for each type crystal, the value of which is highest for Si and the lowest for In-Sb. These respective activation energies are interpreted in terms of the energies required for the formation of defects in the various crystals.

The dynamical behavior of dislocations is profoundly affected by the addition of p or n-type impurities to germanium. A mechanism to account for the impurity effects, involving the generation of defects during the motion of dislocations has been proposed, and is based on the electrical behavior of vacancies. Predictions from this model have been tested and verified in "dislocation-free" germanium, in wh[illegible]ch vacancies behave as acceptors. On the other hand, in silicon for example, the addition of p-type impurities does not influence the dynamical behavior of dislocations.

Since the electrical behavior of defects such as vacancies in silicon and the semiconductor compounds is not known, it is suggested that some knowledge of their behavior can be obtained from experiments, such as the above, on the dynamical behavior of dislocations in the doped "dislocation-free" crystals.

Mechanical Properties of Single Crystals of Silicon:
W. D. Sylwestrowicz, Bell Telephone Laboratories.

Single crystals of silicon with different orientations were tested in the range of temperature from liquid nitrogen to 1400°C. Observed mechanical properties are related to the number of grown-in dislocations rate of straining and oxygen content. Operating slip systems were determined and the mechanism of fracture and upper yield point are discussed.

Temperature and Strain-Rate Dependence of the Upper and Lower Yield Stress in InSb:
M. S. Abrahams and W. K. Liebmann, Radio Corp. of America.

Single crystals of InSb were deformed in tension, the tensile axis being parallel to [145]. The tests were performed at temperatures between 300°C and the melting point (525°C) and at shear strain-rates varying from 1.94×10^{-4} to 1.94×10^{-3} sec^{-1}.

The stress-strain curves are characterized by the appearance of a definite upper and lower yield point, τ_U and τ_L respectively, at all temperatures and strain-rates. The stress corresponding to the first deviation from linearity, τ_I, is observed to decrease linearly to zero stress at the melting point. The upper and lower yield stresses also exhibit a linear temperature and vanish at the melting point. At constant temperature, both τ_I and τ_U increase with increasing strain-rate until a saturation stress is reached. Thereafter, τ_I and τ_U are constant and independent of the applied strain-rate. However, τ_L is observed to be independent of strain-rate for all temperatures and strain-rates employed. The dislocation density is found to be a function of the plastic shear strain only; no dependence on temperature or strain-rate is observed.

The results indicate that the deformation behavior of these crystals is determined by the Peierls-force. In the unsaturated portion of the τ_I (or τ_U) vs. strain-rate curves, the velocity of a dislocation is a function of the stress acting on the dislocation. The Peierls-force establishes the relationship between velocity and stress in this region. In the saturated portion of the τ_I (or τ_U) vs. strain-rate curves, the velocity is no longer a unique function of the stress. Rather, once the saturation stress is reached the velocity of the dislocation can adjust itself to the requirements of the applied strain-rate without a further increase in stress.

The dislocation velocity vs. stress relationship has been determined from the fact that at the upper yield point the applied strain-rate is matched by the plastic deformation of the specimen.

Plastic Deformation of InSb Single Crystals and the Resulting Changes in Electrical Properties:
H. C. Gatos and Mary C. Finn, Massachusetts Institute of Technology.

Plastic deformation of InSb has been studied by bending single crystal bars of suitable orientation to a varying radius of curvature at elevated temperatures. Because of the crystallographic polarity of InSb along the <111> directions the rows of atoms associated with edge dislocations of opposite sign are not the same. Thus, if positive dislocations have a row of In atoms, then negative dislocations have a row of Sb atoms and vice versa. Excess of either type of dislocations can be present according to the orientation of the deformed crystals. These dislocations are identified by etching techniques. The different effects of the two types of dislocations on the electrical properties of InSb will be discussed.

Analysis of Electrical Properties in Plastically Deformed InSb:
J. J. Duga, Battelle Memorial Institute.

In investigations of the electrical properties of plastically deformed InSb, analysis of the experimental data indicated that acceptor and donor sites were created in almost equal densities during uniaxial compression. It was assumed that this resulted from the introduction of vacancies and interstitials, although no detailed models were outlined to substantiate this hypothesis. It will be shown that a more reasonable argument suggests that the changes in electrical properties are a result of the creation of vacancies in the individual subiattices of the compound, and that all the electrical properties (mobility, Hall coefficient, and magnetoresistance) can be interpreted semiquantitatively by using this model.

It will also be shown that the previous analysis of electrical properties in bent specimens of germanium is valid in InSb only for those cases where the scattering by ionized impurities is insignificant. The theory is extended to the case where impurity scattering becomes important (for impurity concentrations greater than 10^{14} cm^{-3} in InSb at 77°K). It will be shown that the neglect of the effects of impurity scattering results in predicted values of the dislocation density which are larger than would be expected from etch-pit counting or from Nye's bending formula.

Finally, the relations between the electrical properties and structural changes as revealed by metallographic observations will be shown to be consistent with the mechanisms associated with dislocation motion and polygonization.

This research was supported by the Office of Naval Research.

Hot Extrusion and Rolling of Silicon:
C. L. Kolbe, General Electric Co.

The commercial deformation of silicon into sheet has not been possible due to its inherent brittleness. Silicon has previously shown some ductility at slow strain rates at temperatures above 1000°C, and with this information in mind samples of high purity zone melted silicon were extruded and rolled from 1300 to 1350°C into thin sheet.

The samples were encased in molybdenum jackets to support and maintain temperature control. Reductions in area of 65 pct from single extrusions and 21 pct from single rolling passes were obtained. Small sheet samples as thin as 0.020 in. were rolled having crack-free sections.

WEDNESDAY, MARCH 1, PM

MBD-EMD-ISD
Basic Science Symposium on Kinetics and Design (II)

(Sponsored by Basic Science Committee of Minerals Beneficiation Division)

2:00 pm Chase-Park Plaza Hotel Starlight Roof

Chairmen:
P. L. de Bruyn, Massachusetts Institute of Technology
R. J. Charles, General Electric Co.
M. E. Wadsworth, University of Utah

Conditioning Rate Studies of an Iron Ore:
E. F. Young, Jr., and J. W. H. Chi, Jones & Laughlin Steel Corp.

A new approach was taken to the study of conditioning of iron ores for flotation. An attempt was made to consider conditioning as a unit operation and establish the controlling mechanism and kinetics. While the complete mechanism proved too complex for detailed analysis, a workable correlation of the kinetics of conditioning was developed. The use of an analog computer in evaluating kinetic constants was demonstrated.

A general type of empirical equation relating reagent level, conditioner size, agitation intensity, and conditioning time to flotation performance is presented. The empirical model is shown to be compatible with theoretical considerations.

Alloy-Salt Exchange Reactions:
A. T. Morkel and T. B. King, Massachusetts Institute of Technology.

The kinetics of the reactions of lead, zinc cadmium and tin, dissolved in bismuth, with bismuth chloride dissolved in a salt melt have been studied by direct rate measurements. It is shown that the data are consistent with rate control of the over-all reaction by either the transport of the solute in the alloy, or by transport in the salt melt, experimental conditions being chosen to favor one or the other of these processes. It is also concluded that the exchange reaction at the interface is fast. Since such reactions would involve the simultaneous collision of several atoms, the over-all reaction probably occurs as the sum of two electro-chemical partial reactions. The apparent activation energies are somewhat larger than might be expected for a purely transport controlled process. It is also shown that the usual methods of establishing forced convection in high temperature systems lead to diffusion boundary layer thicknesses which are relatively constant.

Kinetics of Reduction of Magnetite With Hydrogen and Hydrogen-Water Vapor Mixtures:
J. M. Quets, M. E. Wadsworth, and J. R. Lewis, University of Utah.

Samples of synthetic magnetite were reduced at various temperatures in various partial pressures of hydrogen and in mixtures of hydrogen and water vapor. The reaction was found to be surface controlled in all cases. In the absence of water vapor, the rate was directly proportional to hydrogen partial pressure. The reduction is believed to involve a surface reaction followed by surface diffusion to the reduced bulk phase being formed. A transition in the mechanism occurred at approximately 590°C. This has been attributed to a change in the slow step of the reduction reaction resulting from the presence of stable wustite at higher temperatures. Below 590°C the heat of activation for the reduction was found to be 14,700 cal per mole and 3,200 cal per mole above. A similar mechanism is proposed for the reduction of magnetite to wustite. The retarding effect of water vapor can be quantitatively explained in terms of a back reaction involving the surface diffusing species.

Kinetics of Flotation:
O. F. Devereux and P. L. de Bruyn, Massachusetts Institute of Technology.

Studies aimed at an understanding of the kinetics involved in flotation have in the past consisted mainly of fitting experimental data to empirical relations. A notable exception to this approach is the theoretical analysis by Sutherland.

Dimensional analysis may also be applied to the flotation system in an attempt to obtain an empirical relation between a reduced number of dimensionless parameters. This has been done and a functional relationship relating recovery to five dimensionless groups has been obtained with the restriction that geometric similarity be observed and that the chemical conditions be invariant. Preliminary experimental studies of a simple flotation system indicate that this relationship may be of value in the scale-up of laboratory tests to plant operations.

A theoretical kinetic study is more satisfactory in that it is derived from basic principles and does not suffer from the restrictions that plague an empirical study. Flotation can be described by three steps: (1) collision, (2) adhesion, and (3) levitation to the froth and subsequent froth drainage. Each of these steps must be studied separately and then combined to form an expression for the rate at which the entire process occurs.

The collision rate of colloidal particles as influenced by hydrodynamic flow was treated by Müller. This theoretical analysis of Müller with slight modification may be applied to the collision of particles and bubbles in some flotation systems.

Adhesion of particles to bubbles may be attributed to the combined influence of hydrodynamic forces, Brownian motion, electrical double layer interaction, and the London-van der Waals forces. Preliminary calculations indicate that an analytic description of this process is possible.

No solution has been attempted of the foam drainage problem.

All-Institute Annual Banquet and Reception

7:00 pm Chase-Park Plaza Hotel Khorassan and Regency Rooms

Joseph L. Gillson, President, AIME, Presiding.

THURSDAY, MARCH 2, AM

EMD
Ceramics

(Sponsored by EMD Refractories Committee)

9:00 am Ambassador Hotel Empire Room (Lobby Floor)

Chairmen:
H. R. Hanley, Missouri School of Mines.
W. F. Rappold, General Refractories Co.

Ceramics for Nuclear Power Applications:
L. Ploetz. A. T. Muccigrosso, and R. E. Mistler, General Electric Co.

Internal Stresses in Ceramic Bodies:
R. M. Fulbrath, University of California.

Characteristics of Cerium Oxide Glass Polishes:
L. K. Duncan, Vitro Chemical Co.

IMD
Direct Observation of Imperfections in Crystals II: Optical and Electron Microscopy

(Sponsored by IMD Chemistry and Physics of Metals Committee)

9:00 am Ambassador Hotel Coronation Room (Lobby Floor)

Chairmen:
J. H. Wernick, Bell Telephone Laboratories
R. A. Swalin, University of Minnesota

On the Formation of Dislocations Around Precipitate Particles of Cu_2O in Cu:
F. W. Young, Jr., Oak Ridge National Laboratory.

Single crystals of 99.999 pct copper with (111) faces exposed were carefully annealed and the dislocation content determined by an etch pit technique. Precipitates of Cu_2O were formed in these crystals by oxidizing them in 10μ of O_2 at 900°C, and then furnace cooling. The external oxide and some of the copper was removed by dissolving in HNO_3. After electro-polishing and etching with the dislocation etch, small "islands" of high dislocation content were observed, by use of the optical microscope. Often a small precipitate of Cu_2O could be seen in the center of the island of dislocations. It was determined that this phenomenon occurred throughout the body of the crystal. Measurements were made of the size of the oxide precipitate particles compared to the extent of the dislocation islands around them. Assuming that the dislocations were formed and moved by the stress arising from the precipitate, and that the distance which dislocations were observed from the precipitate was a measure of the stress, the above measurements were analyzed using the theory of Eshelby[2] on the stress produced when a cavity is filled with a particle larger than the cavity. The results suggest that probably the stress associated with a precipitate particle was too large to be accounted for by considering only the difference in coefficient of thermal expansion of the Vu vs. Cu_2O. Rather, they suggest that the stress probably was associated with the conversion of a small volume of Cu to Cu_2O.

Positive and Negative Dislocations and Dislocation Multiplication in Copper:
J. D. Livingston, General Electric Co.

It has been found possible to distinguish between positive and negative edge dislocations in copper crystals by an etch pitting technique. Dislocations of different sign are revealed by etch pits of different depth, which are easily distinguishable in the microscope. The evidence that these pits correspond to dislocations of opposite sign, and the implications of this surprising observation on the mechanism of etch pit formation, will be discussed.

Different mechanisms of dislocation multiplication are expected to lead to different distributions of positive and negative dislocations. Since one now has a technique capable of determining the sign of individual dislocations, one can gain new information on

the processes of dislocation multiplication. Observations on dislocation sources and dislocation multiplication in copper crystals will be presented.

Observations of Imperfections on Thermally Etched Copper Single Crystals:

J. Chirigos, Westinghouse Electric Corp., E. R. Parker, University of California, and M. T. Simnad, General Dynamics Corp.*

Single crystals of copper, in the form of electropolished flat plates having facets within a few degrees of the (111) plane, were placed close together (within one mil distance) and thermally etched in an argon atmosphere at 1000°C. Unusual surface etch patterns were developed, which consist of arrays of concentric, circular steps connected by wavy lines. Distinct jogs are seen throughout this substructure. The jogs form the sites for the initiation of surface reactions such as corrosion and electrochemical exchange with ions in aqueous solutions. These patterns have been shown to continue to some depth in the crystals since they are readily reproduced after repeated electropolishing and thermal etching in an argon atmosphere that is saturated with copper vapor. No such effects could be obtained on single crystals having facets parallel to the (110) or (100) planes. Thermal etching in vacuum did not give rise to these effects.

The imperfections that have been observed are described in terms of a dislocation model and the interaction of stable facets with the initial orientation of the crystal surface.

Observations of Imperfection Effects on the Nucleation and Growth of Nickel Metal in the Reduction of NiO by Hydrogen:

M. T. Simnad, General Dynamics Corp., and A. Spilners, Carnegie Institute of Technology.

The kinetics and mechanism of the reduction of nickel oxide by hydrogen has been shown to be a structure dependent phenomenon, which is influenced by the presence of imperfections in the oxide lattice. The initiation of the reduction process is governed by the crystal structure of the nickel oxide, by the presence of distorted regions in the oxide, and by the temperature.

The nickel oxide was prepared by the oxidation of nickel specimens in oxygen at 1000°C. Some of the specimens were only partly oxidized while others were oxidized completely. The oxides were annealed at 1000°C in vacuum, in argon, and in an oxygen atmosphere. Various types of substructures were produced in the oxides when annealed in the different ambient atmospheres.

The oxides were exposed to a stream of hydrogen gas at temperatures ranging from 250°C to 600°C. At temperatures up to 400°C an induction period was observed, the length of which decreased as the temperature was increased. The nucleation and growth of metallic nickel islands on the surface of the oxides commenced at the end of the induction period, and took place at specific imperfections on the oxide surface. A striking feature of the reduction process at temperatures below 400°C is the nucleation and growth of metallic nickel islands within the body of the oxide. These submerged islands of nickel form at the metal-oxide interface in the partially oxidized specimens, and they appear in the center of the fully oxidized specimens. At temperatures between 400° and 500°C the metal is formed on the surface of the oxide only, and has severe defects. At temperatures above 500°C the metal forms as a uniform, continuous band at the surface of the oxide. These observations are interpreted in terms of the effects of imperfections on surface reactions and of diffusion processes in the oxide and in the metal.

Dislocations and Plastic Deformation in Titanium Dioxide:

W. M. Hirthe, N. R. Adsit, and J. O. Brittain, Northwestern University.

An etch pit technique has been developed for revealing dislocations in TiO_2. The etchants used range from acids such as orthophosphoric, sulfuric, and hydroflouric to fused salts of the alkali metals. A chemical technique for polishing surfaces other than cleavage planes prior to etching has also been developed. The etch pits produced have a size and shape dependent on the character of the dislocation and the particular etchant employed.

Evidence is offered to show a one-to-one correspondence between the pits and dislocations in "as grown" TiO_2 by means of pit patterns on matching cleavage planes, existence and densities of low angle intersecting boundaries, and redistribution and annihilation of pits as the result of an annealing treatment.

The etch pit technique has been applied to investigating the plastic deformation of TiO_2. Dislocation rosettes were produced on several crystal planes by impacting with small steel balls. Slip elements were identified by means of these rosettes and other dislocation arrays introduced in bending and compression. The variation in dislocation density from the neutral axis to the surface of a deformed beam is shown. Annealing of the impact rosettes and dislocation arrays in deformed structures results in polygonization. Additional information regarding dislocation behavior in TiO_2 is presented in terms of the orientation, spacing, and density of polygon walls.

Use of the Field Emission Microscope in the Study of Screw Dislocations:

R. W. Strayer, L. C. Crouser, and J. K. Trolan, Linfield Research Institute.

The use of pulsed field emission microscopy in the study of individual screw dislocations is discussed. The advantages of the method are: (1) the sample may be heated so that the kinetics of processes involving screw dislocations may be followed, (2) the movement of only a few atoms can be monitored, and (3) the stress on the sample to be studied due to the application of a microsecond voltage pulse has negligible effect upon the dislocations within the sample. In this report the method has been applied to tungsten only, but could be used on other materials from which field emission cathodes can be made.

A screw dislocation intersecting a low emission plane on the surface of a field emitter manifests itself as a spiral in the emission pattern; the spiral is due to emission from the step edge produced by the intersection of the screw dislocation with the plane. Step edges of one atom layer in height are resolvable.

If a field emitter is heated, material is removed from the emitter tip under the action of surface tension forces. On low emission planes containing no dislocation intersections, the removal of material appears in the emission pattern as collapsing rings of emission; each collapsing ring corresponds to the removal of one atom layer from the particular plane. For a plane containing a screw dislocation intersection, the removal of material is indicated by the spiral revolving about the point of intersection. When a counteracting electrostatic stress that is greater than the stress due to surface tension is applied to the emitter surface, no ring phenomena are observed, due to the difficulty of nucleating new atom layers upon a crystal plane. However, on a plane containing a dislocation intersection, the spiral is observed to rotate in a direction that indicates the addition of material to the plane, resulting in growth normal to the plane.

The direction of a screw dislocation in a tungsten field emitter has been determined by comparing the step height it produced on one of the {110} planes of the emitter surface to the known distance between atom layers for the [110] direction, and noting the direction of motion of the intersection in the emission pattern. The step height measurement was made by counting the number of spiral revolutions as a function of the number of collapsing rings on another plane of the {110} family. The direction of the screw dislocation was determined to be one of the <111> directions.

Certain bright spots in the high emission areas of the patterns have been correlated with screw dislocation intersections with the emitter surfaces by observing their movement into a low emission area as the emitter surface recedes due to dulling. In the low emission region the previous bright spot becomes the end of a spiral. A count of the number of bright spots in the pattern gives the minimum number of dislocations intersecting the cathode surface, which with a knowledge of the surface area leads to a lower bound on the value for dislocation density.

The support of the Office of Naval Research for this work is gratefully acknowledged.

Deformation of Metals and Alloys by Ultramicrotomy:

V. A. Phillips, General Electric Co.

Slices of metals, only a few hundred angstroms thick, have been cut with a diamond knife in an ultramicrotome. The defects produced have been studied by transmission electron microscopy. Microtoming involves a bending operation as the slice is removed, in addition to a cutting or parting operation. Five main types of crystalline imperfection are produced by microtoming: 1) dislocations 2) dislocation networks 3) dissociated dislocations 4) fault bands 5) ultramicrokink bands.

Individual dislocations are observed in all metals cut, although in metals of low melting point they are mostly arranged in low angle boundaries probably due to rapid recovery occurring at room temperature. Dislocation networks are commonly observed throughout higher melting point metals such as copper; in lower melting point metals they are fragmentary or absent. This is believed due to recovery. Widely dissociated individual dislocations comprising Shockley partials connected by stacking fault are observed in some grains in metals such as silver and copper, particularly when second phase particles are present. Fault banding is a common feature in certain grains of most microtomed face centered cubic metals and alloys. Fault bands are a form of slip band and consist of trains of widely dissociated glissile dislocations, either on a single slip plane or on a group of parallel planes. Hexagonal metals such as zinc become kinked during microtoming and exhibit ultramicrokink bands which resemble previously known phenomena such as Gilman's μ-bands but are three orders of magnitude finer in scale.

Role of Lattice Defects in the Precipitation of Carbon in Alpha Iron:

E. Smith, Department of Mines and Technical Surveys, Canada.

Iron sheet 0.005 in. thick containing about 0.02 pct carbon was quenched from 720°C and aged at 100°C or 200°C. The samples were thinned electrolytically and examined by transmission in the electron microscope.

It was found that the carbon precipitated first as a metastable phase in the form of plates growing on the {100} planes of the iron. On further ageing cementite nucleated and grew in the form of bundles of rods on the {110} planes of the iron while the metastable phase dissolved.

Lattice defects appear to play an important part in the precipitation of the carbon. On ageing at 100°C the first metallographic evidence of the precipitation is the appearance of small loops on the {100} planes of the iron. These are very similar in appearance to collapsed vacancy loops seen in other metals. Their orientations in the foils are consistent with those of collapsed vacancy loops on the {100} planes of the iron, with Burgers vectors <111>. The loops are also seen along the dislocations which were present in the metal after quenching, although it is not clear how the loops are attached to these dislocations. The loops appear to be the sites on which platelets of the metastable carbide grow in the {100} planes of the iron. The growth of the precipitate is more rapid on the loops attached to the dislocations than on the separate loops, suggesting that the dislocations act as channels for diffusion of carbon. If the density of the quenched-in dislocations is very high nearly all the precipitate forms on the loops attached to them.

Dislocations also appear to be important in the nucleation of cementite since this carbide appears always to nucleate at the quenched-in dislocations on which the metastable carbide has precipitated. As the cementite grows the metastable carbide disappears from the dislocations. No cementite particles have been seen which did not have dislocations connected with them.

Dislocation Arrangements in Alpha Iron During Deformation and Recovery:

A. S. Keh, U. S. Steel Corp.**

Dislocation arrangements in a decarburised vacuum-melted iron deformed by rolling and annealed at various temperatures were studied by transmission electron microscopy.

In specimens deformed at room temperature, dislocations were usually joggy and non-uniformly distributed. As the amount of strain was increased, a cell structure was developed. The average dislocation density increased with increasing amount of strain, while the cell dimension remained constant within the range investigated. A linear relationship was found between the square root of dislocation density and flow stress. This result is consistent with the jog theory of work hardening recently proposed by Hirsch and Mott for fcc metals. Piled-up groups of dislocations were very seldom seen.

For the same amount of deformation, the dislocation arrangement changed with deformation temperature. As this temperature was decreased from 250°C to 25°C and then to −70°C, the tendency for cell formation lessened and the dislocation distribution became more uniform.

During recovery, the first noticeable structural change was the straightening of dislocations. This was accompanied by a sharp drop in flow stress, but by only a slight decrease of dislocation density. At a later stage of recovery, dislocations rearranged themselves into regular sub-boundaries and continuously decreased in number, whereas the flow stress remained constant.

Electron Microscope Examination of Crystals of Some Inorganic Compounds:

R. W. Horne, M. McMorris, and R. H. Ottewill, Cavendish Laboratory, England.

The crystals which we examined are silver iodide, cuprous iodide and lead iodide. Ageing of nuclear silver iodide sols, under controlled conditions, leads to the formation, primarily, of thin flat hexagonal crystals of width 5,000-20,000 Å and of thickness 300-700 Å. On formation, the crystals of cuprous and of lead iodide are already sufficiently large and no ageing is required. They attain widths of 30,000 Å and 200,000 Å respectively, and are mostly thin flat plates. These crystals have been found suitable for direct electron microscope examination of interior changes occurring within them during irradiation. Micro-diffraction has shown that the silver iodide crystals are in the hexagonal phase with a (001) orientation: that the cuprous and lead iodide crystals are fcc and hcp structures with (111) and (0001) orientations, respectively.

During irradiation of silver iodide crystals with the electron beam, filaments are observed to grow from some of the side faces and corners of hexagonal prisms and from the apices of tetrahedra. Micro-diffraction has shown them to consist of silver. Striations, whose interpretation is not yet quite clear, appear frequently on these filaments at an angle to the geometric axis. The growth may be explained by the motion of a screw dislocation through the body of the crystal; it is possible that, in some cases, migration of silver towards the apices of tetrahedra might be the operating mechanism.

In all three kinds of crystal, rapid migratory changes of contrast may be produced under conditions similar to those producing filament growth. These might, possibly, be interpreted as voids. Extensive networks of dislocations and several single dislocations and their migration have been observed in lead iodide; in silver and lead iodide, dislocation loops, twins and stacking faults. All these observations are possible only after a certain level of illumination has been exceeded and are due to thermal gradients which are set up in the crystal. Stacking disorder should occur readily in silver iodide since the lattice energies of the two low-temperature phases (Wurtzite and ZnS structures) are almost equal.

In silver iodide, dislocation loops are in greater evidence when the crystal has entered the b.c.c. α-phase (stable above ~ 146°C.), in which phase voids and twins may be observed also. The orientation of this phase is (110) and twinning has been observed to take place on two sets of (112) planes resulting in twins with (110) and (001) orientations.

Interpretation of Transmission Electron Microscope Images of Dislocations:

A. Howie, Cavendish Laboratory, England.

Dislocations in thin foils are made visible in the transmission electron microscope because of the altered conditions of Bragg diffraction in the strained crystal near the dislocation. The diffraction scattering can be described by coupled differential equations which relate the amplitudes of the various transmitted and diffracted waves at a given depth in the foil. These equations take account not only of dynamical diffraction processes, but also of important anomalous absorption effects which depend on the crystal orientation and are also influenced by lattice strains. The equations have been integrated numerically using a high speed digital computer and theoretical image profiles obtained corresponding to dislocations in various situations.

Many of the results of a simple kinematical diffraction theory already published are confirmed. In particular, the importance of the extinction distance, ξ_g, in fixing the image width and the value of $g \cdot b$ (the scalar product of the dislocation Burgers vector b and the diffraction vector g) which determines the nature of the image are now well established. With these results it is now possible to determine Burgers vectors and analyze dislocation interactions in favorable circumstances. It is also possible to determine the sense of Burgers vectors and hence to tell whether dislocation loops are of the vacancy or interstitial type and whether stacking faults are intrinsic or extrinsic.

Some of the complicated dotted and zig-zag dislocation image effects which were not understood on the basis of the kinematical theory are explained by the more exact dynamical theory and shown to be a diffraction effect and not a consequence of any structural feature of the dislocation. The dynamical theory has also been used to explain the image effects observed at edge dislocations with Burgers vectors normal to the foil. A further important prediction of the dynamical theory concerns the relation between the bright field and dark field images and is of considerable practical importance in deciding which part of a dislocation or stacking fault is near the top of the foil and which part near the bottom of the foil.

It should be possible to use the theory to investigate the electron microscope contrast effects from many other crystal defects. Important information about the diffraction and absorption of electrons in crystals may also result from such investigations.

Observations of Dislocation Arrangements in FCC Single Crystals of Different Stacking Fault Energy:

A. Howie, Cavendish Laboratory, England.*

Direct estimates of stacking fault energy in a number of solid solution alloys (Cu + Al, Cu + Zn, Ag + Al, Ag + Zn) have recently been made from electron microscope observations of extended dislocation nodes. In the case of the Cu + Al alloys, the stacking fault energy, γ, falls from an estimated value of ~ 40 ergs/cm² for pure Cu to a value of only ~ 2 ergs/cm² for Cu + 15 wt. pct Al.

The author has prepared single crystals of a number of Cu + Al alloys of different composition, strained them as bulk specimens, and examined the internal dislocation arrangement as a function of deformation.

In the easy glide region of the stress-strain curve almost all the dislocations seem to lie on the primary slip plane and dislocations of opposite Burgers vectors are frequently found in neighboring regions.

The linear hardening stage of deformation is characterized by the onset of slip on other systems leading to dislocation reactions some of which have been identified. In pure Cu and in the alloys where $\gamma \gtrsim 20$ ergs/cm², this leads to a very irregular dislocation arrangement with a rough cell structure in which it is impossible to distinguish the slip planes on which the dislocations lie. In the alloys of low stacking fault energy, piled up groups of a very relaxed type are found. In no case is the arrangement of dislocations in accord with the predictions of the pile-up model of work hardening where long range stresses are assumed.

Stage III of the deformation is characterized in Cu and the alloys of higher stacking fault energy by the appearance of increasing misorientations between different regions, many of which appear to be separated by twist boundaries on either the primary or secondary slip plane. In the alloys of lower stacking fault energy wide stacking faults and occasionally twins are formed on these planes.

The interpretation of these results in terms of current theories of work hardening in fcc single crystals will be discussed.

Dislocations in Layer Structures:

S. Amelinckx and P. Delavignette, Center for the Study of Nuclear Energy, Belgium.

The layer lattices have recently provided interesting subjects for the study of dislocations. Specimens for transmission electron microscopic studies can easily be prepared by simple cleavage. The main glide plane in general being parallel to the cleavage plane, the dislocation arrangements are almost exclusively in the plane of observation. In a number of cases the stacking fault energy is small and visible dissociation into partial dislocations results. In these cases a direct determination of the stacking fault energy is possible, either from a measurement of the distances between partials, or from the radius of curvature of an isolated partial. The determination of the Burgers vector is much facilitated because one plane in which the Burgers vector is lying is known a priori.

The following layer structures have been investigated:

1) Antimonium- and bismuth telluride, bismuth selenide and their alloys.
2) Silicate layer structures: talc, pyrophylite, muscovite, chlorite.
3) Graphite and the structurally related molybdenum disulfide.

A survey will be given of the results of the investigation, and the observations in graphite, talc and molybdenum disulfide will be discussed in detail.

A. Graphite: The dislocations consist of wide ribbons of stackings bounded by partials. The stacking fault corresponds to one layer in rhombohedral stacking. Assuming a reasonable value for μ, the stacking fault energy is found to be 3-5 ergs/cm². The interactions between these ribbons of partials and the formation of networks will be discussed. Quenched and irradiated specimens will also be discussed.

B. Molybdenum disulfide: The stacking energy is very small for dislocations in one lattice plane and larger for those in the second type of lattice plane. Patterns rather similar to those in graphite on the one hand and to those in bismuth telluride on the other hand are usually found.

C. Talc: The dislocations in this material form wide ribbons consisting of four partials separated by stacking faults; the stacking fault energy can be evaluated from the geometry of these ribbons.

Typical features observed in the other layer lattices will be discussed, as well as some contrast effects observed in these crystals.

Decoration of Dislocations and Low Angle Grain Boundaries in Graphite Single Crystals:

R. Bacon and R. Sprague, Union Carbide Corp.

Dislocations and low angle boundaries in single crystal graphite have been decorated by introducing Cs, Br, and $FeCl_3$, which are known to form lamellar compounds with graphite. The existence of pockets or internal cracks bounded by tilt boundaries has been inferred.

Decorated dislocations have been observed in the electron microscope as fine lines of precipitated impurity along directions parallel to other, undecorated dislocations. Very frequently it is found that such a line forms a boundary between a region which is dotted with precipitated impurities, and a region which is perfectly clean. It thus appears that the dislocation had moved across its slip plane and swept the impurities along with it. Apparently, the dislocation then freed itself of the impurities, for within clear regions of frequently occurring breaks along the line of precipi-

tate, the dislocation could not be found; this in spite of tilting the specimen, and in spite of the visibility of other dislocations in the region.

The existence of internal cracks between layers has been inferred from several types of observation. In one type, a liquid-like turbulent motion of the impurity substance is observed within a well-defined grain. Such grains are commonly observed in these studies, and probably result in most cases from sample preparation, i.e., are due to crinkling of the foil. Their boundaries are thus low angle tilt boundaries or perhaps two closely spaced high angle tilt boundaries. The turbulent motion is very definitely confined within the boundaries of the grain, and this is taken as evidence that these boundaries are indeed tilt boundaries representing bends in the layers, and acting as effective barriers to crack propagation.

The existence of long ribbon-like internal cracks, bounded on one or both sides by low angle tilt boundaries, has been inferred from the decoration of such boundaries. Stroh has worked out the theory of formation of such cracks in materials which, like graphite, cleave on their slip planes, and they have been observed in zinc by Gilman. In the present study we have seen decorated grain boundaries in which the decoration is asymmetrical with respect to the center of the boundary. This is believed to be due to the crack propagating away from the tilt boundary on one side only. Frequently such decorated boundaries occur in closely spaced parallel pairs in which the pattern of decoration of one is the reflection of the other. This suggests that it resulted from a second tilt boundary which terminates the crack on the opposite side.

Finally, there exists evidence that condensed vacancy disks can act as incipient pockets or circular bubbles within which gases or liquids may be confined. We frequently observe both condensed vacancy disks in their flat or collapsed state, and also bubbles of very similar size (the "Hohlstellen" described in Liesegang). In one instance such a bubble was observed to expand and contract and finally "explode" due to beam heating.

This research was sponsored in part by The Air Research and Development Command and The Air Materiel Command, United States Air Force.

Dislocations and Plastic Flow in Molybdenum:
G. Thomas and R. B. Benson, University of California.*

Well-annealed polycrystalline sheets of molybdenum have been deformed in tension at room temperature by various amounts to fracture. Thin foils were then prepared from these by electropolishing in sulphuric acid and examined in the Hitachi H. U. 10 electron microscope. Even after as little as 1 pct strain, many dislocation networks were observed, and from selected area diffraction work it has been deduced that these form mainly from screw dislocations of a/2 [111] Burgers vector interacting in (110). The networks then consist of screws of Burgers vectors a/2 [111], a/2 [1$\bar{1}$1], a [010] at the nodes. These results indicate that the onset of plastic flow is accompanied mainly by the movement of screw dislocations. As the deformation was increased, the dislocations formed into well-defined tightly knit networks or sub-boundaries (e.g. 3 pct strain). Finally after the maximum strain (5 pct), the whole structure consisted of an intimate tangle of dislocations which were difficult to resolve.

Dislocations did not move under the influence of the electron beam, which suggests that they are pinned, probably by interstitial solute atoms. By carrying out strain-ageing experiments, it was possible to decorate some of the dislocations with precipitates and this confirms the hypothesis of pinning. During recovery at about 300°C the dislocation networks broke down, forming many dislocation loops. These probably result from the climb of dislocations.

Stacking faults were never observed in the specimens; neither were the dislocation nodes extended. This leads to the conclusion that molybdenum has an intermediate or high stacking fault energy.

Work hardening in molybdenum is thus attributed to the interaction between dislocations and the networks, and to the formation of immobile segments in sub-boundaries.

More recent experiments involving deformation at other temperatures, and further work on the recovery of cold worked molybdenum will be described.

* To be read by title only.
** To be read by title only, oral presentation Tuesday am.

EMD Zinc Round Table Discussion

(Sponsored by EMD Lead, Zinc, and Tin Committee)

9:00 am Ambassador Hotel Embassy Room (Lower Level)

Chairman:
R. L. Hennebach, American Smelting and Refining Co.

Round Table Discussion on Removal of Lead From Zinc Concentrates Prior to Retorting for the Production of Continuous Galvanizing Alloys:

Moderator:
N. E. Cook, Wheeling Steel Corp.

Participation by:
M. E. Hughen, American Metal Climax Co.
K. H. Schultz, American Smelting and Refining Co.
N. S. Geist, American Zinc, Lead and Smelting Co.
H. D. Carus, Mathiessen-Hegeler Zinc Co.
J. S. Van Aken, National Zinc Co.
John Winters, St. Joseph Lead Co.

THURSDAY, MARCH 2, AM

ISD Steelmaking

9:00 am Ambassador Hotel Versailles Room (First Floor)

Chairmen:
F. J. McMulkin, Dominion Foundries and Steel Co.
T. E. Dancy, Jones & Laughlin Steel Corp.

Desulfurization in the Open Hearth:
T. B. Winkler, Bethlehem Steel Corp.

Basic Oxygen Process, Thermodynamics or Equilibrium Achieved:
H. W. Meyer, Jones & Laughlin Steel Corp.

Ladle Vacuum Degassing With Bottom Bubbling-Pilot Plant Results:
J. Duflot, J. Verge, and Etienne Spire, Institut de Recherches de la Sidérurgie, France.

Progress Report on the Oxygen-Lime Powder Process:
B. Trentini, P. Vayssiere, and C. Roederer, Institut de Recherches de la Sidérurgie, France.

Collecting of Unburnt Gases From Oxygen Converters:
M. Allard, P. Vayssiere, and G. Namy, Institut de Recherches de la Sidérurgie, and Cie. des Ateliers & Forges de la Loire, France.

IMD Structure and Properties

(Based on submission of research abstracts)

9:00 am Ambassador Hotel Marquis Room (First Floor)

Chairmen:
E. E. Underwood, Battelle Memorial Institute
I. S. Servi, Kelsey-Hayes Co.

X-Ray Studies of the Structure of Electro-Deposited Silver:
L. H. Schwartz and J. B. Cohen, Northwestern University.

Measurements of stored energy and tensile strength of electrodeposited silver have suggested that the plate is in a state of disorder much like that of severely cold-worked silver. To examine the plated structure, X-ray diffraction studies were made of samples prepared by plating from an aqueous solution of KCN-AgCN on both sides of annealed silver strips. Diffraction patterns were analyzed by the Fourier methods described by Warren and Averbach.

Observations of the effect of current density, and the addition of the brightening agent CS_2 will be reported. A detailed Fourier analysis has been made of the diffraction patterns from samples plated at 3°C, 29°C, and 60°C. Current density was held constant and plate thickness fixed at a value greater than "infinite thickness" for CuK_α radiation. The particle size and root-mean-square strains will be discussed. An analysis for faulting will also be presented. These observations are correlated with the stored energy measurements of Schottky and Bever.

This research was supported in part by ARPA.

Effect of Heat Treatment on the Structure, Mechanical Properties, and Stress Corrosion Resistance of Heavy Forged Sections of Zircaloy-2:
J. H. Schemel, The Carborundum Metals Co.

Large Zircaloy-2 hammer or press forged bars are machined into end caps for nuclear fuel elements. Material forged into large bars for this service did not meet the requirements for corrosion resistance in 750°F steam. Weight gains of coupons cut from the forged bars were 40 to 60 mg/dm² compared to the specified maximum of 38 after exposure to steam for 14 days at 750°F and 1500 psi.

The mechanism of this corrosion and its relation to microstructure is discussed. Based on these observations, a solution heat-treatment followed by rapid cooling was tried on coupons from eight heats of forged bars. The basket-weave structure resulting from the quench did not show agglomeration of intermetallic compounds. These coupons gave uniformly good corrosion resistance with weight gains very close to the 28 mg/dm² expected of Zircaloy-2 after the 14-day stream test.

A large forging was solution heat-treated and tested for structure, corrosion resistance, and mechanical properties. Corrosion resistance was excellent and uniform throughout the section. The normally anisotropic mechanical properties were changed to completely isotropic. Strength levels were raised while there was a small loss in ductility at the service temperature. Tempering of the quenched structure below the beta transus did not improve the ductility at the test temperature at 600°F.

Alternate forging practices that may also improve corrosion resistance are discussed.

THURSDAY, MARCH 2, AM

Effect of Composition on the Properties of 5 pct. Cr Steels:

E. G. Schempp, Atlas Steels Limited, and W. A. Morgan, Department of Mines and Technical Surveys, Canada.

The influence of modifications in the molybdenum, vanadium and, to a limited degree, carbon and boron content to a basic composition of 5 pct Cr, .75 pct Mo, 1 pct V hot-work tool steel composition, was studied in respect to hardenability, grain coarsening temperature and tempering characteristics. Tensile, impact and fatigue properties were studied for the most promising compositions. An alloy with 0.40 pct C, 5 pct Cr, 2.25 pct Si, 2.25 pct Mo and 1.00 pct V was found to offer the most favorable combination of properties in respect to high strength applications when tempered at 1100°F.

Several Factors Affecting High-Temperature Strength of Cermet:

Kaizo Monma and Hajime Suto, Tohoku University, Japan

The cracks formed in cermet at high temperature were observed mainly at interfaces between hard compound and metal or at grain boundaries of compound. Therefore, it is considered that the interface or grain boundary play an important role on the high temperature properties of cermet. Of course, a cermet is an agglomeration with hard particles in a softer matrix, therefore the strength of compound, the strength of binder material and the state of aggregation must be considered as the important factors influencing the strength of cermet. Then, the state of aggregation may be controlled not only by the amount of binder and the initial particle size of powders, but also by the solubility and the wettability of the cermet system. An aim of this paper is to clear that the contributions of these various factors to the strength of cermet, and to give a theoretical picture for the investigaion of cermet. The contents of this paper are as follows:

(1) In order to prepare a compact cermet, the contact angle between the binder metal and the hard compound is required at least less than 90°. For understanding the general tendency of the contact angles, the contact angles between liquid metals (copper, silver, nickel and some other metals) and various refractory compounds (silicides, borides, carbides and nitrides) were measured. It is shown from these data that in general, wettability closely depends on the relative positions in the periodic table of molten metal and elements consisting of the refractory compound. For example, the degree of wetting of Ti-compounds by a molten metal increases in the order of $TiO_2 \rightarrow TiN \rightarrow TiC \rightarrow TiB_2 \rightarrow Ti_5Si_3$, and the wettability on TiC increases in the order of Zn → Cu → Ni → Co → Fe → Mn → Cr → V, and the degree of wetting of carbides by copper increases in the order of B_4C → Graphite → SiC → TiC → VC $Cr_3C_2 \rightarrow Fe_3C$.

(2) Even if a contact angle is very small, when the solubility of a compound in a binder is too high, a cermet with fine structure can not be prepared by liquid sintering. Then the solubilities of some compounds in liquid copper and liquid nickel have been measured. As the result, it was shown that the smaller the contact angle of the system is, the more the solubility increases.

(3) The energies of surface and of interfaces play leading roles for the contact angle or the wettability, while the bulk energies relate to the solubility of compound in metal. Based on these conclusions, an alloying element in binder metal may affect the contact angle differently from the solubility. Then the solubility and the contact angle of the systems (TiC, Cr_3C_2) – (Cu – Ni) were measured. For example, in TiC-Cu system, it was shown that the contact angle decreases rapidly with increasing nickel content in copper, while the solubility of TiC in liquid copper increases gradually. In general, it seems that the curves of the contact angle and of the solubility vs. the concentration of alloying element in binder metal are convex downwards individually. In order to prepare a cermet with fine structure, it is very beneficial to use an alloy-binder containing a small quantity of good-wettable metal.

(4) The cermets manufactured by a same process with a same size of powder and a same amount of metal, may have various structures depending on the difference of wettability and the solubility between compound and binding metal. The above mentioned distinction of structure may affect the strength of the cermet. In order to discuss the relationships, the transverse rupture strengths of various compounds-copper (or nickel, and copper-nickel alloys) cermets were investigated, and the structure and the mode of cracks caused by tests were observed microscopically. From these experimental data, it was shown that the controlling factors of strength of cermet are not only the strength of compound and of the binder metal itself, but also the state of aggregation in the structure. Consequently, the wettability and the solubility of a cermet system have strong influences on the strength of the cermet.

(5) Of course, the state of aggregation may be controlled by varying the amount of binder and the initial grain size of powders. In succession to (4), the effects of grain size and the amount of binder on the strength of cermet were ascertained in detail with the WC-Co system.

(6) Besides the above-mentioned experimental results, to determine the high temperature strength of cermet, the following equation is deduced theoretically:

$$S = kTE\alpha/\beta \ (\log t - \log hn/ZkT - \Delta F/kT)$$

where, S is the stress applied, k the Boltzman's constant, T the absolute temperature, E the Young's modulus, t the time to rupture, n the number of atoms participated in the formation of cracks, Z the number of atoms at the sites of stress concentration, h the Plank's constant, ΔF the sum of the activation energy of self-diffusion and the surface tension per atom, α and β are constants concerning the stress relaxation and some other factors. On the basis of the experimental data obtained, the above theoretical equation is discussed, and at least qualitatively, shows good agreements with experimental results.

Resoftening of Quench-Hardened Aluminum:

T. Mori, M. Meshii, and J. W. Kauffman, Northwestern University.

Resoftening of quench-hardened aluminum wire with purity of 99.999 pct was studied. Specimens which had a diameter of 0.016 in. were quenched from 600°C into iced brine and annealed at temperatures between 100°C to 260°C after aging at room temperature for several hours. After this annealing treatment, the yield stress was measured at room temperature. The obtained results are as follows: When the annealing time was 30 min, the annealing temperature at which half of the increase of hardening by quenching and aging was eliminated was 200°C, and more than 260°C was necessary to anneal the specimen into fully softened condition. Isothermal annealings which were performed at 170°C to 230°C showed that the activation energy for resoftening was about 1.2 eV. The temperature dependence of the yield stress was higher for the quenched specimens than for the annealed ones.

Neither the rate nor the temperature of resoftening corresponds to the disappearance of prismatic dislocation rings as observed by electron microscopy. Therefore, prismatic dislocation loops may not be a major cause of quench-hardening in aluminum. The suggested mechanism of quench-hardening in aluminum will be presented in terms of dislocation configurations in connection with supersaturation of vacancies.

This work was supported by the Office of Naval Research.

Silicon and Titanium Interactions in a Ni Cr Fe Mo Heat-Resistant Alloy System:

E. F. Becht and G. S. Ansell, Rensselaer Polytechnic Institute.

The effect of silicon variations in the heat resistant alloy system 12 Cr, 42 Ni, 6 Mo, balance Fe is studied at four titanium levels. Mechanical properties, particularly stress rupture life and room temperature tensile ductility, are seen to be extremely sensitive to silicon content at all titanium levels studied. The progressive deterioration of mechanical properties with increasing silicon is accompanied by the precipitation of a grain boundary structure. Isothermal aging studies in conjunction with charpy impact measurements are used to map the embrittling reaction for all combinations of titanium and silicon investigated. This reaction is shown to be independent of the age hardening (gamma prime) reaction.

Titanium increases in the range 2.36 to 3.30 pct produce, at constant silicon levels, progressive strength increases up to 3.10 pct Ti. Above this titanium level secondary precipitation is observed even at the lowest silicon levels studied and no further strength increases are noted.

Radiation-Induced Overaging of a 2024-T3 Aluminum Alloy:

A. M. Adair, R. E. Hook, and H. J. Garrett, Aeronautical Research Laboratory, Wright Patterson Air Force Base.

While conducting research for the Aeronautical Research Laboratory, Patterson, Moos Research reported that a 2024-T3 aluminum alloy severely overaged when subjected to a total reactor irradiation dosage of 8.5×10^{19} nvt (fast). The hardness of this alloy decreased from an initial value of 165 VHN before irradiation to a value of 60 VHN after irradiation. The microstructure of the irradiated alloy was described as showing a large amount of grain boundary precipitate pius fine precipitate evenly distributed throughout the grains. The question was raised as to whether the overaging and attendant loss of mechanical strength was due to neutron irradiation or simply to temperature, since the temperature during irradiation was not measured and the microstructure is very similar to alloys of this type which have been heated to a temperature near 350°C.

The results of the present investigation have shown that the observed overaging was due to the irradiation environment and not due to temperature alone. Aging experiments on both irradiated and unirradiated samples of this alloy were followed by hardness measurements, metallographic examination and an analysis of X-ray back reflection patterns. These experiments included samples of lower total radiation dosage. It was determined that the temperature of the alloy could not have been above 200°C for the total time of irradiation, which is an insufficient temperature to cause the severe overaging. An unirradiated sample of the alloy which was successively annealed for 10-day periods at 50°C intervals between 250°C and 350°C, possessed a final microstructure and X-ray back reflection pattern almost identical to the irradiated one. Since the aging process is dependent upon a vacancy controlled diffusion mechanism, it is concluded that the radiation induced overaging was primarily due to an increase in the vacancy concentration of the alloy caused by neutron bombardment. The data obtained indicate that the irradiation dosage received by this aluminum alloy is equivalent energywise to an increase in temperature of approximately 150°C.

Strain Aging of α-Iron:

E. P. Lautenschlager, Allis Chalmers Manufacturing Co., and J. O. Brittain, Northwestern University.

An investigation was conducted on fine grained alpha-iron wire to measure the influence of the temperature of deformation on strain aging near room temperature. Specimens were deformed various amounts at 77°, 195°, 273°K; aged; and the strain aging followed by either internal friction or return of the yield point measurements. The results show that strain aging occurs in three stages. In the first stage the concentration of solute atoms decreases as measured by internal friction methods while a small yield point appears that increases in magnitude to about 2 pct, during this stage the solute concentration appears to remain constant. In the second stage the solute concentration decreases while the magnitude of the yield point increases. In the third stage the solute atoms continue to drain from the solution, the yield points approach a maximum and the strain hardening coefficient and flow curves increase. Low temperature anneals at 77° and 195°K did not influence the kinetics of the strain aging reaction.

The kinetics of the strain aging reaction were not the same for the two sets of measurements, the internal friction results lag behind the yield point measurements. However, strain aging kinetics at 44° and 64°C showed no effect due to the difference in deformation temperature.

This research was supported by the U. S. Air Force through the Air Force Office of Scientific Research of the Air Research and Development Command.

Some Effects of Adsorbed Polar Molecules on the Properties of Ionic Crystals:
A. R. C. Westwood, Research Institute for Advanced Studies.

Studies have been made of some effects of adsorbed polar molecules on the mechanical behavior of lithium fluoride and potassium chloride crystals. Specimens were tested in compression and bending, crystal surfaces being either chemically polished or polished and then sprinkled with carborundum powder to introduce dislocation half-loop surface sources.

It was observed that adsorption of, for example, stearic acid molecules from a hexadecane solution tended to prevent the operation of such artificially-introduced surface sources so that the yield stress of sprinkled crystals was raised to that of polished crystals—in bend tests an increase of some 85 pct. The subsequent rate of work hardening of sprinkled crystals was reduced by the presence of adsorbed polar molecules (Rebinder-effect) provided that the concentration of the surfactant in the inert testing medium was sufficiently low.

An explanation for these and other observations will be offered based on considerations of the preferential adsorption of polar molecules at the points of emergence of dislocations and at kink sites and on the resulting effects on dislocation motion and multiplication.

This research was partly supported by Watertown Arsenal Laboratories.

Effect of Hydrogen on the Mechanical Properties of Columbium:
T. W. Wood and R. D. Daniels, University of Oklahoma

The mechanical properties of high purity columbium containing quantities of occluded hydrogen have been studied. Hydrogen was introduced into the metal at elevated temperatures by a thermal equilibrium technique. A series of hydrogen-columbium alloys containing up to ten atomic pct hydrogen were prepared.

Strength of the alloys did not depend appreciably on hydrogen content, but ductility as determined by reduction in area in a tensile test was quite sensitive to hydrogen content. A ductile-to-brittle transition was observed at about room temperature for alloys containing one atomic pct or more hydrogen. This transition temperature increased with hydrogen content. The reduction in area of columbium in a tensile test at −321°F was 75 pct, compared to 3 pct for a 1 pct hydrogen alloy. The embrittlement was reversible, since a vacuum outgassing treatment restored the unhydrogenated ductility. Embrittlement decreased with increasing temperature. At 400°F alloys containing as much as 6 atomic pct hydrogen failed with greater than 97 pct reduction in area.

Hydrogen drastically altered the mode of failure. Pure columbium failed by shear on one or more planes at 45° to the tensile axis. In hydrogenated columbium multiple planes of separation were observed, both parallel and normal to the tensile axis. At room temperature the fracture process was silent, even for apparently brittle samples. This is explained by the concept of separation across hydride platelets in an inherently ductile matrix. The mechanical agent for crack formation is believed to be segregation of hydrogen into oriented flakes of an expanded second phase, creating a stress concentration at the flake rim.

This work was sponsored by the United States Atomic Energy Commission.

Structure and Properties of Dispersion-Strengthened Nickel Alloys Produced by Internal Oxidation:
L. J. Bonis and N. J. Grant, Massachusetts Institute of Technology.

Dilute solid solutions of silicon, chromium, titanium and aluminum in nickel were studied to determine the oxide particle size and shape, the interparticle spacing of the oxide, the change of oxide size with depth of penetration, and the rate of oxide penetration, all of these as a function of the kind and amount of solute, and the time and temperature of reaction.

From these studies it was possible to select the optimum conditions of powder diameter, time and temperature to produce completely reacted powders; these powders were then compacted, sintered and extruded into bar stock, providing material for low temperature tensile and high temperature creep-rupture tests.

Studies were also made of the stability (by means of room temperature hardness tests) of these alloys as a function of time at temperature. The better alloys do not lose hardness up to 1300°C, attesting to their remarkable structural stability.

Room temperature tensile strength values of 105,000 psi with 15 pct elongation were achieved. Stress for a 100-hour life values at 815° and 1000°C are about 30 pct greater than the best values previously reported for dispersion strengthened nickel-metal oxide alloys produced by mechanical blending of powders.

EMD
Third Extractive Metallurgy Division Lecture

11:00 am Ambassador Hotel Empire Room (Lobby Floor)

Reinhardt Schuhmann, Jr., Chairman, *Presiding*.

THURSDAY, MARCH 2, AM

Speaker:
Antoine Gaudin, Massachusetts Institute of Technology.

Subject:
Separation Engineering on the Moon.

THURSDAY, MARCH 2, PM

EMD
Application of Refractories in Ferrous and Non-Ferrous Furnace Technology

(Sponsored by EMD Refractories Committee)

2:00 pm Ambassador Hotel Empire Room (Lobby Floor)

Chairmen:
H. H. Hanley, Missouri School of Mines.
W. F. Rappold, General Refractories Co.

Current Status of Basic Brick Technology:
G. R. Eusner, U.S. Steel Corp.

Refractories in Secondary Aluminum Industry:
C. A. Licht, U. S. Reduction Co.

Refractories in Furnaces Copper Refining Industry:
B. J. Lewin and Ralph Wigger, Lewin-Mather Co.

Suspended Roofs, Silica Bottoms, Basic Side Walls, and Uptakes:
F. Richardson and R. Law, Phelps Dodge Refining Corp.

IMD
Direct Observation of Imperfections in Crystals III: Topographical Diffraction Methods

(Sponsored by IMD Chemistry and Physics of Metals Committee)

2:00 pm Ambassador Hotel Coronation Room (Lobby Floor)

Chairmen:
J. B. Newkirk, Cornell University.
B. D. Cullity, Notre Dame University.

X-Ray Diffraction Images of the Domains in $BaTiO_3$:
A. Guinier and M. Lambert, University of Paris, France.

X-ray diffraction images of natural faces of $BaTiO_3$ crystals have been made by the Berg-Barrett technique, the reflecting planes being (100) or (001). While single domain crystals give images of uniform intensity, figures with strong contrast are obtained with polydomains.

There is a striking resemblance between the general appearance of the observed striations and the system of fringes observed with the microscope and polarized light, namely the square fringes described by Forsbergh.

We have shown that the origin of the contrast of the X-ray images is not a difference of the primary extinction from point to point, because the crystals are probably not good enough to have a high extinction in any place.

One finds that the location of the black striations on the X-ray image changes when the orientation of the crystal is slightly changed. This indicates that the variations in the reflected intensity are due to variations of the orientation of the reflecting planes. Successive photographs allow a detailed map to be made of the local deviations of the crystallites. The result, in the case of one type of $BaTiO_3$ crystal, is that the planes, theoretically parallel to the surface of the crystal, are in fact dome-shaped,

each dome being inscribed in a square, the edges of which appear clearly in the micrograph with polarized light. The angles of the reticular planes along the edges are of the order of a few tens of minutes.

We are now trying to relate these irregularities with the texture of the domains suggested by the optical observations. The influence of the temperature and of an electric field on the microtexture revealed by X-ray images is being experimentally studied and we hope that some results can be given at the meeting.

Determining Recrystallization by a Diffractometer Technique:

C. S. Barrett, University of Chicago.

The progress of recrystallization of cold-worked metals can be detected by the growth of sharp spots in Laue or Debye diffraction patterns, or of reflections from strain-free grains recorded on X-ray reflection micrographs. A related method that has some valuable characteristics is based on the use of a counter diffractometer instead of a photographic film. The counter is set in a stationary position on one of the strong Debye rings and the specimen is then rotated or oscillated a few degrees and the diffracted intensity recorded on a chart. The beginning of recrystallization is recognized by the appearance of small peaks from the recrystallized grains superimposed on the relatively smooth record from the cold-worked matrix, and by a drop in intensity of the background between the peaks.

The method was applied to sodium and to zone-refined bismuth during heating at one to two degrees per minute and yielded precision of the order of +2°C in the temperature of beginning recrystallization. The dependence of this temperature on the temperature of cold work was studied in bismuth. The temperature range involved was below that which is accessible to metallographic methods.

Professor Jin-ichi Takamura and the author have found that some information can also be obtained on rates of grain growth of individual recrystallized grains, and rates of disappearance of cold-worked material, though high precision in these measurements is not claimed.

Several X-Ray Methods for Observing Defects in Crystals:

G. Borrmann, Fritz-Haber-Institut der Max-Planck-Gesellschaft, Germany.

First Method: Wide-angle photographs
Results of transmission photographs of calcite plates up to six centimeters thick; characteristic: layered structures. Silicon photograph in the growth direction.
Second Method: Fine beam encompassing a large volume of the crystal by the beam.
Photographs of silicon normal to the growth direction; characteristic: definite defects in dislocation-free region.

Direct Study of Imperfections in Nearly Perfect Crystals:

R. H. Bragg, Armour Research Foundation, and L. V. Azaroff, Illinois Institute of Technology.

The Laue focussing geometry employed by Guinier and Tennevin to observe imperfections in deformed crystals can be used to study imperfections in nearly perfect crystals as well by interpreting the observed intensity distributions in terms of the dynamical theory of x-ray diffraction. To do this, a suitable oriented single crystal is irradiated by a polychromatic x-ray beam emanating from a virtual point source. The x-rays diffracted by a family of planes are detected by a proportional counter and their intensity and energy distribution is measured by a multichannel pulse-height analyzer and scaler. The direct beam incident on the crystal is similarly analyzed so that the ratio I/I_0 can be directly and accurately measured. The measured integrated intensity can then be interpreted directly in terms of imperfections producing a "mosaic" character in the otherwise perfect crystal by utilizing the extinction formulas of the dynamical theory of x-ray diffraction. The utilization of a point source of x-rays permits the examination of very small regions of the crystal without introduction of spurious effects due to collimation, etc..

A rather perfect silicon crystal was thus examined. It was found that neighboring regions of the same crystal slice consisted of "mosaic" blocks approximately 10^{-5} cm on edge whose relative tilts ranged from 5 to 15 seconds of arc. As suggested by the more qualitative etch pit densities and diffraction line widths, it was found that the central portions of the crystal contained larger blocks with smaller relative misorientations than those lying near the surfaces of the crystal. It is thus possible to use this method to map accurately the distribution of imperfections throughout as small or as large a region of a single crystal as is desired.

An incidental by-product of this investigation was a quantitative evaluation of the dependence of x-ray perfection studies on the wavelength of the x-radiation employed. This method becomes insensitive to the small deviations from perfection studied for wavelengths greater than 1.18Å and increases in sensitivity directly as the wavelength is decreased below this value.

This investigation was supported in part by the Office of Scientific Research of the United States Air Force.

X-Ray Picture of the Field of Lattice Distortions Around a Single Dislocation:

U. Bonse, Physikalisches Institut der Universitat Munster, Germany

On x-ray micrographs taken by the double-crystal method, dislocations seldom become visible as mere shadows. Mostly there is seen at the place of the dislocation a peculiar profile of x-ray intensity very typical for the dislocation concerned. This profile often shows a black-white structure. In many cases it can be detected up to a distance of 50 microns from the dislocation line.

The origin of this phenomenon may in the first place be sought in the field of the elastic lattice distortions around a dislocation. In continuation of previous investigations the intensity profile has been more exactly analysed in order to find out the relation between x-ray intensity and lattice distortions. For this purpose a double crystal spectrometer of high thermal and mechanical stability was used. X-ray intensity was measured by high resolution photographic films in connection with the application of a microphotometer especially constructed to resolve details of less than about 1 micron apart.

The intensity profile around a dislocation was found to depend not only on the choice of the plane of reflection but also on the position of the crystal within the range of reflection. With x-rays belonging to the middle of the range a dislocation and its surroundings reflect *less intensively* than undeformed parts of the crystal: this means that dislocations are visible as shadows. X-rays from either side of the middle of the range of reflection produce distinct black-white profiles at dislocations, the profiles having different sign on different sides of the middle of the range of reflection. Finally, with rays from the borders of the range, dislocations reflect *more strongly* than the rest of the crystal. Within limits of measurement, the maximum observed reflection power does not exceed that of an ideal crystal. This suggests that probably it is not reduction of primary extinction but rather misalignment of the deformed parts of a crystal with respect to the condition of reflection that makes dislocations visible with the double crystal method. Local twist as well as local strain of the lattice may be acting here. In support of this argument some examples are given, where calculated intensity profiles qualitatively match with observed ones. Here it is assumed that local twist and local strain are the only causes of intensity changes.

Furthermore the quantitative measurement of x-ray intensity in the vicinity of a dislocation gives some aspects to the problem of x-ray diffraction by a weakly deformed lattice.

X-Ray Diffraction Microscopy With the Use of Crystal Monochromator:

K. Kohra, University of Tokyo, and M. Yoshimatsu and I. Shimizu, Rigaku Denki Co., Japan.

In X-ray diffraction microscopy by use of chraacteristic X-rays, the dispersion of wave length, especially $K\alpha_1$ and $K\alpha_2$ radiations, limits the resolution of the image. The following countermeasures have been hitherto taken:

i. In order to make the separation of the image due to the doublet as small as possible, the plate is put as close as possible to the specimen, as in the Berg-Barrett technique and techniques depending on abnormal transmission;

ii. A narrow incident beam is used and the $K\alpha_2$ radiation is excluded with slit system. In order to obtain a photograph from an extended region, specimen and photographic plate are driven at the same time *in* a certain direction. This is the so-called Lang camera.

As another technique, we use a monochromater of highly perfect crystal in the + position for the specimen. A diffraction micrograph with a highly monochromatized beam is obtained. This technique is applied to reflection as well as transmission photographs.

A double crystal monochromater may also be used. In this case the cause of the image contrast is different from in the former case. In the former case, the extinction effect is utilized, while in the latter case the change of the reflection position or the decrease of the reflection percent is utilized.

The cause of the image contrast in the technique of Bonse and Kappler is the same as the latter case we have described. However in their technique the disperison of the doublet is not avoided in the photograph.

X-Ray Diffraction Topographic Studies of Dislocations in Germanium and Floating-Zone Grown Silicon:

A. E. Jenkinson, Mullard Laboratories and A. R. Lang, Bristol University, England.

Dislocations have been studied by the method of projection topographs in a single crystal of silicon grown in vacuo by the Keck method. The crystal was p-type with average resistivity about 1,000 ohm cm. A number of slices was cut from the crystal and at least three stereo pairs of projection topographs was taken of each slice. Burgers vectors and the spatial configuration of dislocations could thus be determined. The majority of dislocations appeared to have been generated by plastic deformation with sources in or near the crystal surface. Much dislocation movement, particularly climb, had taken place behind the solidification front, making it difficult to identify genuine "grown-in" dislocations. The effect on dislocation contrast of elastic deformation of the crystal will be illustrated, and a comparison made between the quality of dislocation images in this crystal and in crucible-grown silicon crystals.

Germanium crystals grown by the floating crucible technique have also been examined. These ranged from dislocation counts of 10^6 per cm^2 down to practically dislocation-free material. A variety of curious diffraction effects has been observed due to the relatively high absorption of the X-rays in germanium. The progressive reversal of dislocation image contrast with increasing thickness has been followed. Some low-angle boundaries have been observed and in certain cases it has been possible to resolve the individual dislocations in a low-angle boundary.

X-Ray Diffraction Microscopy of Silicon Single Crystals:

G. H. Schwuttke, General Telephone and Electronics Laboratories, Inc.

X-ray diffraction microscopy has been used successfully to reveal dislocations in single crystals. In this paper its application in the detection of impurities in the semiconductor lattice will be described. Segregation, cluster formation and precipitated second phases can be seen very clearly in the diffraction image.

Segregation effects in silicon were studied in crystals rotation-grown in a quartz crucible. Such crystals are known to exhibit microsegregation of oxygen, which occurs in planes perpendicular to the growth direction.

Microsegregation of oxygen is found to produce a strong variation of the diffracting power and, accordingly, can easily be detected in the diffraction image. The sensitivity of this method is comparable to that of the 9 μ absorption method. The oxygen can be detected in crystals before any heat treatment and also after treatment at 1000°C.

Precipitation effects were also studied in silicon crystals containing copper precipitates, since in this case X-ray results can be verified by infrared microscopy; good agreement was observed. Resolution in the X-ray image is good enough to produce a picture that is identical with that obtained by infrared microscopy.

The research reported in this paper was sponsored by the Electronics Research Directorate of the Air Force Cambridge Research Center, Air Research and Development Command.

Direct Observation of Dislocations in Germanium by an X-ray Method:

V. Gerold and F. Meier, Max-Planck-Institut fuer Metallforschung, Germany.

The anomalous transmission of X-rays in nearly perfect crystals (Borrmann effect) makes it possible to see single dislocations. A crystal slice is placed in the Bragg position for a set of lattice planes going through the slice. According to the dynamical theory, wave fields are formed within the crystal. Their energy is flowing parallel to the reflecting planes having an anomalous low absorption coefficient. The strains around a dislocation increase the absorption coefficient. In this way a shadow of the dislocation may be seen.

Using crystals having a high normal absorption coefficient such as germanium, a simple experimental arrangement can be used. With this technique it is possible to get transmission pictures of an area of 1 cm^2 in two hours, using filtered copper radiation. The method is useful for crystals having a dislocation density lower than 10^4 lines/cm^2.

Germanium crystals (20 x 6 x 1 mm) with a low dislocation density have been deformed in compression at about 650°C in an atmosphere of purified hydrogen. In our first experiments the entire crystal was held at a temperature of 650°C and compressed. The dislocations that were introduced during the deformation came from the areas near the anvils of the compression device.

In order to eliminate the effect of the anvils, a second series of experiments has been performed using a special furnace in which the ends of each crystal are held at about 150 to 200°C while the central region is heated to the plastic region. In these experiments the additional dislocations originate on the crystal edges in the heated region. Dislocation loops originating at the crystal surface have been found, but are fewer in number.

Direct Observation of Anti-phase Domains of the Ordered Alloy Cu_3Pd by Means of Electron Microscope:

S. Ogawa and D. Watanabe, Tohoku University, Japan.*

The anti-phase domain structure of the ordered Cu_3Pd alloy was previously studied by electron diffraction using thin films, and the domain structure was concluded to be two-dimensional, unlike the one dimensional structure in CuAu (II) or Ag_3Mg. In the present work two-dimensional domains of Cu_3Pd have been observed directly on single-crystalline evaporated films by means of transmission electron microscopy. Split superlattice reflections, $(010)_{cub}$ and $(110)_{cub}$ groups, have been used to form images, the dark field technique being adopted. Two side lengths of the two-dimensional domain in several alloy films have been estimated from electron diffraction patterns to be 12 to 16Å and 21 to 27Å, respectively. Several systems of parallel lines have been observed on electron-microscopic images, and there are two definite values of the line interval which well coincide with the two side lengths of the domain estimated by electron diffraction. Crossing lines which have been obtained by using the $(010)_{cub}$ group reveal most intuitively the two-dimensional domain.

It has been concluded from distribution of various line systems that six orientations of the domain structure which were assumed in the structure analysis of the previous electron diffraction study take place certainly in the alloy film. Thus, the two-dimensional structure of Cu_3Pd has been confirmed by means of electron microscope. Explanations have been given also to oblique lines and double intervals.

X-Ray Diffraction Micrography of Aluminum Oxide Single Crystal:

J. Lommel and M. Kronberg, General Electric Co.

When certain geometrical conditions are satisfied to obtain high resolution, the Berg-Barrett X-ray technique can be used to observe the dislocation structure of crystals. Dislocations are observed by the reduction in primary extinction which their strain fields cause. This results in a local increase in reflecting power of X-rays from a crystal. By observing at high magnification the X-ray reflection from a crystal, the arrangement of individual dislocations can be seen. The best results in observing dislocations have been obtained in materials of low atomic number, such as Al, LiF, and Al_2O_3.

A detailed study has been made of single crystal of aluminum oxide with the corundum structure grown by the Verneuil flame fusion process. The surface studied was 28° from a prism face. It was prepared by grinding with diamond powder and then by heating to a temperature where evaporation of the surface occurred, removing the surface scratches. A line focus X-ray source was used to irradiate a large area of the crystal and to obtain a resolution of 3 microns. Chromium K_α radiation was used.

Misorientations between subgrains ranged up to 2°, with most boundaries having a misorientation of several minutes. In some subgrain boundaries the misorientation was small enough so that individual dislocations in the boundary could be resolved. Two sets of dislocations were observed. In one group the dislocation lines were perpendicular to the subgrain boundaries but were lined up parallel to one another in a band which was parallel to the subgrain boundaries. By observing reflections from several sets of planes and noting the reflections in which the dislocation contrast reversed, the Burgers vector was deduced to be $\frac{1}{3}[\bar{2}110]$. The second group consisted of single or groups of dislocations which were near the surface and were perpendicular to the subgrain boundaries. The contrast effects observed with this set of dislocations were also consistent with a $\frac{1}{3}[2\bar{1}10]$ type of Burgers vector. The dislocation density within the subgrains was of the order of 10^4 per cm^2.

Hardness impressions were made in the sample at room temperature. The X-ray reflections from this heavily deformed region consisted of a central, non-reflecting spot where the misorientation was too great to obtain a reflection, and a highly reflecting region with a four-fold symmetry around the central spot. No dislocations were observed to move out of the strained region. The highly reflecting region is one where either there are elastic strains or there are many dislocations spaced too closely for the resolution of the technique to be observed as individual ones.

Study of Dislocation Arrays by X-ray Diffraction Microscopy:

W. H. Robinson, Carnegie Institute of Technology.

The work to be reported on was initiated for the purpose of developing the technique of x-ray diffraction microscopy as a means of studying dislocation arrays, primarily in metal crystals, in order to determine dislocation interactions during deformation. A comparison of the results obtained by diffraction microscopy, the Lambot Laue reflection method, and thermal etching of some silver single crystals of (001), (110), and (111) orientations was made. Dislocation densities obtained from the Lambot method are only semi-quantitative, while those obtained from counting thermal etching pits and from the x-ray diffraction microscopy are felt to be quantitative. The dislocation arrays observed thus far are essentially random as found in as-grown crystals and are the order of 10^7 per cm^2.

Chromium radiation has been used for the reflection from both silver and copper crystals and good resolution has been obtained. A discussion of the conditions for good resolution will be given.

This research was supported by the United States Air Force through Air Force Office of Scientific Research.

Substructure and Dislocation Networks in Tungsten:

S. Weissmann, Y. Nakayama and T. Imura, Rutgers University.

The stubstructure and dislocation network of single crystal and polycrystalline tungsten were studied by means of x-ray reflection microscopy combined with diffraction analysis based on the double-crystal diffractometer principle, as well as by transmission electron microscopy. Various orders of magnitude of substructural entities could be disclosed:

1) a macroscopic substructure with large disorientation angles,
2) a microscopic substructure contained in the macroscopic subgrains and characterized by smaller disorientation angles,
3) dislocation networks and stacking faults within the subgrains.

The substructure and dislocation network of single crystal and of the combination of x-ray reflection microscopy and diffraction analysis the larger order of substructure could be quantitatively studied. Thus, from measurements of the disorientation angles between adjacent subgrains and from measurements of the lattice misalignment within the subgrains, the dislocation densities in sub-boundaries and within the subgrains were estimated.

The smallest order of substructure was disclosed by transmission electron microscpy through the direct observation of the dislocation networks. Specimens subjected to various degrees of deformation and subsequent annealing were investigated. Gradual heating up to 1500°C of a 50 pct deformed specimen gave rise to subgrain growth. During this process the dislocations were swept out of certain subgrains into the subgrain boundaries, reducing drastically the density of dislocations in these subgrains and rendering some nearly dislocation-free. Other subgrains, on the other hand, retained the high dislocation density, which varied between 10^9-10^{10} per cm^2. Sometimes dislocation pockets detached themselves from the boundaries of the growing subgrain and rearranged themselves in such a manner as to form a fine network in the subgrain, which in the meantime had advanced its interfaces during the growth process.

Moire patterns resulting from internal double reflection of the electron beam due to the small disorientation angles of overlapping subgrains were observed preponderantly in the annealed specimens.

At elevated annealing temperatures (1500°C and above) stacking faults were observed exhibiting the typical extinction contrast described by Whelan and Hirsch.

Interesting observations of tree-shaped configurations were made which were interpreted to result from the interaction of dislocations lying on planes.

Grains recrystallized at elevated temperatures from about 800°C upward appeared to be the product of preferential subgrain growth whereby the strain induced growth mechanism played an important part. The precipitation of impurities at grain boundaries at elevated temperatures and the interaction of dislocations with impurities were also investigated.

*Papers to be read by title only.

EMD Extractive Metallurgy of Zinc

(Sponsored by EMD Lead, Zinc, and Tin Committee)

2:00 pm Ambassador Hotel Embassy Room (Lower Level)

Chairmen:

C. H. Cotterill, American Zinc, Lead and Smelting Co.

H. R. Bianco, St. Joseph Lead Co.

THURSDAY, MARCH 2, PM

Continuous Tapping of Metallurgical Furnace:
J. T. Roy, American Smelting and Refining Co.

Possibilities of Isolations and Alterations in the System Lead-Tin-Antimony-Copper:
Josef Blanderer, Rau Metallverk Gesellschaft, Germany.

Concentrating Zinc Ore at Anaconda:
J. A. Stephens, Anaconda Co.

Uses of By-Product Oxygen in Roasting Processes at the Electrolytic Zinc Company of Australasia in Tasmania:
R. W. Pickering and D. J. Gaffney, Electrolytic Zinc Co., Australia.

Use of Oxygen-Enriched Air in Cominco's Metallurgical Operations at Trail, B. C.:
L. Landucci and F. T. Fuller, Consolidated Mining and Smelting Co. of Canada, Ltd.

ISD Blast Furnace Technology

2:00 pm Ambassador Hotel Versailles Room (First Floor)

Chairmen:
H. N. Lander, Youngstown Sheet and Tube Corp.
R. L. Stephenson, US Steel Corp.

Thermal Balance for a Ferromanganese Blast Furnace:
B. M. Shields and W. H. Burr, U.S. Steel Corp.

Kinetics of Reduction of FeO and MnO From Silicate and Aluminate Slags:
S. K. Tarby, Lehigh University, and W. O. Philbrook, Carnegie Institute of Technology.

Reduction Kinetics of Hematite in H_2-H_2O-N_2 Mixtures:
W. M. McKewan, U.S. Steel Corp.

Smelting Taconite Pellets in the Blast Furnace:
W. E. Marshall, Armco Steel Corp.

Injections of Fuel in the Blast Furnace Operations:
E. Noel, J. Molderez, and A. Poos, Le Centre National de Recherches Metallurgiques, France.

IMD Mostly Phase Relations

(Based on submission of research abstracts)

2:00 pm Ambassador Hotel Marquis Room (First Floor)

Chairmen:
R. P. Elliott, Armour Research Foundation.
H. M. Davis, Pennsylvania State University.

Constitutional Diagrams Ta-Ru, W-Ru, Ta-Os:
E. J. Rapperport and M. F. Smith, Nuclear Metals, Inc.

Binary constitution diagrams of Ta-Ru, W-Ru, and Ta-Os are presented. The solvus lines and intermediate phase boundaries of these diagrams were determined using several techniques including lattice parameter extrapolation and electron microbeam probe analysis. The salient features of the diagrams are as follows:

Ta-Ru: Ru is soluble in Ta to approximately 45 atomic pct there are two intermediate phases, one from 50 to 60 atomic pct Ru, and one at 62 atomic pct Ru decomposing peritectoidally at 1667°C; Ta is soluble in Ru to 28 atomic pct.

W-Ru: Ru is soluble in W to 23 atomic pct; a sigma phase forms peritectically from the melt at 40 atomic pct Ru, ~2300°C and decomposes eutectoidally at 1687°C; Ta is soluble in Ru to 48.

Ta-Os: Os is soluble in Ta to 22 atomic pct; a sigma phase forms peritectically at approx 2500°C, its breadth extending from 21 to 43 atomic pct Os; a second intermediate phase is peritectically formed at 2420°C and 37 atomic pct Os; Ta is soluble in Os to greater than 25 atomic pct.

This work was sponsored by Wright Air Development Division, U. S. Air Force.

Solid Solubilities of Carbon in Cu-Pd-Ni and Cu-Pd-Ag Alloys:
M. E. Nicholson, University of Minnesota and K. K. Rao, Northwestern University.

The solid solubility of carbon has been determined in the ternary systems Pd-Cu-Ag and Pd-Ni-Cu at 900°C. The purpose of this study was to determine what the relative influence of the strain energy associated with adding carbon to the lattice and the electronic structure of the solid solution on the solubility of carbon. The solubility of carbon was determined by saturating the samples with carbon at 900°C in an atmosphere of carbon monoxide in equilibrium with graphite. In addition, precision lattice parameter, Hall coefficient, and magnetic susceptibility measurements were made on many of the alloys.

Hall potentials for palladium-silver and palladium-copper alloys were measured, both before and after carburization. Hall coefficients calculated from these data indicate that the carbon solubility is not proportional to the number of holes in the "d" band.

The solid solubility of carbon in palladium-copper-silver and palladium-nickel-copper alloys appears to be primarily determined by the electronic structure of the alloys. The variation in strain energy in these systems appears to have very little effect on the solid solubility. The solid solubility appears to be proportional to the density of states at the Fermi surface of the "d" band.

This research was sponsored in part by the Atomic Energy Commission.

A Study of the Iron-Nickel-Vanadium System:
C. H. Armitage, Allis-Chalmers Manufacturing Co., and D. J. Mack, University of Wisconsin.

The 1000°C isotherm of the ternary alloy system iron-nickel-vanadium was investigated by X-ray and microscopic methods. It was found that the alpha iron, gamma, and sigma fields of the binary systems extended into the ternary system to form a three phase triangle with vertices at (57Fe,15Ni,28V±2 atomic pct Fe,Ni,V), (40Fe32Ni28V±2at.%Fe,Ni,V) and (48Fe,13N,39V±4 atomic pct Fe, Ni±2 atomic pct V). The ternary sigma field connected the iron-vanadium and nickel-vanadium binary systems and appeared to have a slightly greater range of compositional stability than in either of the binary systems. Both single and two phase theta fields disappeared with the addition of less than 5 atomic pct Fe. The boundary of the vanadium-rich solid solution was found to be a straight line which connected the iron-vanadium and nickel-vanadium binaries respectively at approximately 42 and 6 atomic pct Va.

The region separating the vanadium solid solution and sigma fields contained an unknown phase, appropriately distributed, which was tentatively indexed on a tetragonal unit all with parameters a=2.961A and C=8.922A. It is possible that this cell may be regarded as a three-high stacking of the bcc vanadium cell with nickel and iron atoms in two of the three body centered positions. On this basis the saturated six atom cell should contain 5/6 V in the nickel-vanadium system, and 4/6 V in the ternary system, which was in good agreement with the experimental findings. Significantly, the previously reported NiV_3 phase did not appear, and it is suggested that the unknown phase, rather than NiV_3, forms peritectically at 1380°C.

Investigation of the Systems $CuInTe_2$, $AgInTe_2$, and $AuInTe_2$:
S. M. Zalar, Raytheon Manufacturing Co., and I. B. Cadoff, New York University.

The systems $CuInTe_2$, $AgInTe_2$ and $AuInTe_2$ were investigated as part of a broad program of study of semiconductors of the chalcopyritic structure $A^{I}B^{III}C_2^{VI}$. Thermal, metallographic, mechanical and electronic methods were used. $CuInTe_2$ and $AgInTe_2$ were found to be congruent melting and single phase, exhibiting extrinsic semiconductor properties. The composition $AuInTe_2$, which solidified via a peritectic type reaction, was two phase and metallic in character. Alloys of the form $Ag_xAu_{(1-x)}InTe_2$ and $Ag_yCu_{(1-y)}InTe_2$ were also studied. In the case of Ag-Au substitutions all alloys prepared were two phase and metallic. On the other hand Ag-Cu substitutions resulted in a series of solid solutions over the entire range as indicated by lattice parameter, melting range and thermoelectric power data.

This work was supported by Aeronautical Research Laboratory, WADC.

Tungsten-Oxygen System:
W. T. Ebihara, Carnegie Institute of Technology, and M. J. Pool, G. R. St. Pierre, and Rudolph Speiser, Ohio State University.

The standard free energy of formation of WO_2, $W_{18}O_{49}$, $W_{20}O_{58}$, and WO_3 have been determined in the temperature range of 700 to 1200°C by direct equilibration with H_2-H_2O and CO-CO_2 gas mixtures. The $\Delta F°_T$ data may be summarized as follows:

$W + O_2 = WO_2$ $\quad \Delta F° = -134{,}000 - 17.54T + 179.4T$
$2.778\ WO_2 + O_2 = 2.778\ WO_{2.72}$ $\quad \Delta F° = -124{,}000 + 33.51T$
$11.111\ WO_{2.72} + O_2 = 11.111\ WO_{2.90}$ $\quad \Delta F° = -151{,}350 + 65.42T$
$20\ WO_{2.90} + O_2 = 20\ WO_3$ $\quad \Delta F° = -151{,}800 + 69.47T$

A tentative tungsten-oxygen phase diagram is presented based on the evidence that $W_{18}O_{49}$ and $W_{20}O_{58}$ are unstable, at one atmosphere total pressure, below 590°C and 480°C, respectively.

The research was supported by the U. S. Air Force and by a Universal Cyclops Steel Corp. Fellowship.

Lattice Constants of Alpha Zirconium Alloys With Additions of Ag, Cd, In, Sn, and Sb:
J. O. Betterton, Jr., and D. S. Easton, Oak Ridge National Laboratory.

X-ray measurements were made of the effects of Ag, Cd, In, Sn, and Sb on the lattice constants of the c.p. hexagonal phase of zirconium. The values can be represented to within about one part in 2000 by the following relations:

$$a = 3.2326 - 0.276x$$
$$c = 5.1473 - 0.44x + 0.181xn - 0.016x^2n$$
$$c/a = 1.5923 + 0.056xn$$

where x and n are the atomic fraction and the valency of the solute in the alloy. The proportionality of the axial ratio to solute valency was found to be principally due to a similar dependence of the c-lattice constant upon solute valence, suggesting a possible electron interaction with the Brillouin zone in the [001] direction in alpha-zirconium. The results are explicable in terms of a rigid band model of the alloy with two bands of electrons; one, a valency s-p type band shared with the solute atoms, and another, a d-type band which is not shared with the solute atoms. Assuming a mechanism such as that proposed by H. Jones, electron additions from all of these solutes would be expected to produce equal shifts in the Fermi surface energy and equal electron "pressures" on the lattice dimensions. Since the c dimensions only are affected by the number of s-p electrons from the solute, the Brillouin zone effect is likely to be in this direction. Further, since each solute electron has an equal effect upon the axial ratio, one might conclude that there are few s-p valency-type electrons in solid zirconium, and that the four outer electrons are of the d-type in the solid metal. The measurements were made by Debye Scherrer film technique using a 11.43 cm Phillips Camera, Nelson Riley extrapolation, and at the temperature 23°C ± 4°C. Wire samples of the alloys were prepared from iodide zirconium and high purity solutes and were vacuum annealed. The a and c dimensions are in Angstrom units assuming the following wave lengths for copper radiation: α_1 = 1.54050A, α_2 = 1.54434A, and β = 1.39217A.

Constitution Diagram Columbium-Rhenium:

B. C. Giessen, R. Nordheim, and N. J. Grant, Massachusetts Institute of Technology.

In the course of a large, cooperative study of the phase diagrams of refractory metals, the Nb-Re diagram was worked out using pure starting materials, careful melting techniques, incipient melting, and metallographic and X-ray methods. The solidus lines and phase boundaries were thus determined. The diagram is characterized by a large niobium solid solution range up to 45.5 atomic percent rhenium, and an intermetallic sigma phase at about 54 atomic percent rhenium, which decomposes eutectoidally into niobium solid solution and the second intermetallic phase, chi. Chi phase has a large range of solubility between 61.5 and 67 atomic percent rhenium, and melts congruently, while sigma decomposes on melting to chi and chi melt. Two eutectics occur between niobium solid solution and sigma, and between chi and rhenium solid solution. The literature on this diagram is reviewed, good agreement with a most recent publication is found to exist, and discrepancies with other authors are discussed.

Constitution Diagram Tungsten-Hafnium:

B. C. Giessen and N. J. Grant, Massachusetts Institute of Technology.

In the course of a large, cooperative study of refractory metals phase diagrams in which the system niobium-rhenium was worked out, the diagram tungsten-hafnium was covered, employing the same techniques of incipient melting observations, metallography and X-ray studies. Solidus lines and phase boundaries were determined. There is a peritectically melting Laves phase W_2Hf, a eutectic between W_2Hf, and β-Hf and an eutectoid decomposition of β-Hf into W_2Hf and α-Hf. The temperatures of all these invariant reactions were established; special care was devoted to outlining the range of stability of β-Hf. The eutectoid concentration is surprisingly low at 94 percent Hf. A recent publication of the diagram is reviewed.

Oxidation of Refractory Metals as a Function of Pressure, Temperature, and Time: Tantalum in Oxygen:

J. N. Ong, Jr., Washington University.

By assuming that the oxide layer formed upon reaction of the refractory metals with oxygen does not control the rate of reaction, an equation describing the linear rate of oxidation has been developed by conventional chemical kinetic theory of the form.

$$\text{Rate} = \frac{d(m/A)}{dt} = A' \exp(-\Delta H^{\ddagger}/RT)\,[O_i]$$

Where m/A is weight per unit area, t is time, A' is a constant, H is the enthalpy of activation, R the gas constant, T the absolute temperature and O_i is the concentration of atomic oxygen interstitially dissolved in the metal in the region of the metal-metal oxide interface.

An expression describing O_i as a function of the experimental variables pressure, p, and temperature, T, has been derived of the form:

$$O_i = C_T x_{oi} = C_T$$

$$\left\{ \frac{\frac{1}{\epsilon}\exp\left(-\frac{\Delta H}{RT}\right)\exp\left(\frac{\Delta S}{R}\right)\exp\left[\frac{W}{RT}(2x_{oi}-1)\right]p^{1/2}}{1+\frac{1}{\epsilon}\exp\left(-\frac{\Delta H}{RT}\right)\exp\left(\frac{\Delta S}{R}\right)\exp\left[\frac{W}{RT}(2x_{oi}-1)\right]p^{1/2}} \right\}$$

Where C_T is a conversion factor relating mole fraction of interstitial oxygen, x_{oi}, to concentration, ϵ is a proportionality factor, ΔH and ΔS are the enthalpy and entropy change associated with solution of gaseous oxygen into the metal lattice w is an interaction energy term.

By treating the diffusion of interstitial oxygen and vacancies into the metal by conventional application of the diffusion laws, an expression for the time dependence on the rate of reaction has been developed of the form:

$$\text{Rate} = \frac{d(m/A)}{dt} = A' \exp\left(-\frac{\Delta H}{RT}\right)^{\ddagger}[O_i]$$

$$\left\{ \operatorname{erf}\left(\frac{t}{\tau}\right)^{1/2} + \left(\frac{\tau}{t_\tau}\right)^{1/2} \exp\left(-\frac{t}{\tau}\right) \right\}$$

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erf is the error function, and $1/\tau$ is a rate constant associated with immobilization of vacancies. τ is given by, $\tau = A'' \exp \frac{\Delta H^{\ddagger\prime}}{RT}$, A'' is a constant and $\Delta H^{\ddagger\prime}$ is an enthalpy of activation. By use of the following values; A' = 7.1 x 10^{16} (mg/cm²hr); $\Delta H^{\ddagger}$ = 58,900 (cal/mole); $1/\epsilon \cdot \exp\left(\frac{S}{R}\right)$ = 2.32 x 10^{-31} (atm)$^{-1/2}$, ΔH = −43,680 (cal/mole); w = 3,380 (cal/mole); $\Delta H^{\ddagger\prime}$ = 15,200 (cal/mole); and A'' = 0.179 (sec), the oxidation behavior of tantalum is adequately described in the temperature range 500 to 1400°C and pressure range 0.2 to 40.8 atm.

Portions of this work were sponsored under a consulting agreement with McDonnell Aircraft Corporation, St. Louis, Missouri.

Transgranular Cracking in Austenitic Stainless Steels:

G. Meyrick and H. W. Paxton, Carnegie Institute of Technology.

Theories of stress corrosion cracking which postulate a mechanical step in the propagation of the crack must explain how a brittle crack can move in a normally ductile alloy. Speculations on this problem have been advanced by Forty and by Paxton and Reed, who considered a fast moving brittle crack entering the ductile alloy from a region embrittled by some electrochemical action. Little experimental evidence is available in support of the existence of a brittle step in stress corrosion cracking. Attempts to promote brittle failure in austenitic stainless steels by experiments designed to test the suggestions of the above authors are described here.

Initially, tensile tests at strain rates up to 10^4/ins/in/sec at both room temperature and −193°C were performed using single crystals of Fe-20 pct Cr-20 pct Ni and polycrystals of Fe-18 pct Cr-8 pct Ni and α-brass. Some of the former specimens were preloaded in boiling $MgCl_2$ so that they contained a few fine stress corrosion cracks. In all cases, no evidence of brittle failure could be detected.

The model envisaged in the theoretical considerations was more closely approached in a second series of experiments. Polycrystalline samples of stainless steel were bonded to Armco iron. In the latter component of each specimen, a fast moving brittle crack was caused to run towards the austenite. Examination revealed that in one case a fine crack of a few thousandths of an inch in length and depth was produced in the austenite. It is possible that this observation supports the suggestions that brittle failure can occur over short distances in austenitic steel.

This research was supported by an Office of Naval Research Contract.

Diffusion in Isoviscous Solutions of Cadmium-Mercury and Zinc-Mercury:

H. A. Walls and W. R. Upthegrove, University of Oklahoma.

Golik and Karlikov have studied viscosity, temperature, structure, and composition in several binary amalgams. Coincident viscosity-temperature curves (20°C to 140°C) were reported for several pairs of Cd-Hg and Zn-Hg solutions, e.g., 13.9 atomic pct Cd in Hg and 8.7 atomic pct Zn in Hg. These pairs are described as isoviscous solutions. X-ray diffraction investigations of these isoviscous amalgams indicated that they also possess coincident maxima in their radial distribution functions.

Self-diffusion coefficients of cadmium and zinc in various isoviscous amalgam pairs are measured as a function of viscosity, temperature, and composition. These isoviscous pairs are established by determining the viscosity as a function of temperature between 40°C and 200°C for several parametric concentration levels. Diffusion data are obtained using radioisotopes of cadmium and zinc with the capillary-reservoir technique.

These data permit a critical evaluation of the validity of the Stokes-Einstein equation (independent of the model from which derived) as used to predict diffusion coefficients in liquid metal systems. Since the solution structures as characterized by the radial distribution functions are similar for isoviscous solutions, diffusion measurements permit reasonable speculation as to the relative influence of structure on diffusion.

This research is sponsored by the U. S. Atomic Energy Commission, Metallurgy and Materials Branch.

MBD-EMD

Pyrolysis and Agglomeration

(Sponsored by MBD Pyrolysis and Agglomeration Committee and EMD Pyrometallurgy Committee)

2:00 pm Chase-Park Plaza Hotel Zodiac Room

Chairmen:

H. E. Rowen, McDowell Co., Inc.

W. F. Stowasser, Jones & Laughlin Steel Corp.

Beneficiation of Cement Raw Materials by Dwight-Lloyd Processes:

C. D. Thompson, D. C. Violetta, and C. A. Czako, McDowell Williams Co.

The mechanics of the continuous sintering process are briefly reviewed and the application of this process for the beneficiation of principal minerals used in making cement is shown. Pilot plant tests using pyrobeneficiation techniques were conducted with mix-

tures of limestone and shale in various ratios. Results are illustrated and show the percentage elimination of carbon, carbonates, sulphur, alkalis and volatiles as a function of the limestone-shale ratio. Tests with varying quantities of solid fuels, added to mixtures containing limestone and shale in a 4 to 1 ratio, were made to attain optimum elimination of the volatile constituents. Experimental results show that 90 pct of these constituents were removed during the calcination sintering process. The advantages of using the beneficiated material for making cement are discussed with regard to preparing, handling, and firing operations.

A sintering process for treating raw materials or kiln waste products which contain high amounts of alkaline materials is described. Chloridizing roasts were performed with a high alkaline waste dust mixed in a 1 to 1 ratio with raw cement meal. The data from these tests and others using different halides for aiding alkali removal are evaluated by the alkali content of the cement clinker. Test results showed that the Dwight-Lloyd process could be applied with facility in beneficiating raw materials for cement making. Substantial quantities of deleterious volatile constituents were removed by calcining and sintering techniques.

Exothermic Hardening of Copper Nickel Sulfide:
M. P. Sudbury and F. Petkovich, Falconbridge Nickel Co.

The paper describes the development of a new method of treating flotation concentrates for the preparation of feed suitable for direct charging to blast furnaces or converters. The method takes advantage of the fact that the pyrrhotite contained in the concentrates can be made to undergo exothermic reactions which, under properly controlled conditions, result in the formation of oxidation products serving to cement the concentrate particles together. Thus green concentrates either in the form of pellets or briquettes containing 5 to 8 pct moisture were not only hardened, but also completely dried autogenously by continuous counter-current shaft treatment with air. The process was developed through pilot plant operations ranging from 35 lbs/hour to 2.5 tons/hour, culminating in product evaluation tests on a railway car scale.

New Process in Sintering of Fine Iron Ores:
Y. Takahashi, M. Serizawa, K. Miyagawa, and Y. Shimomura, Fuji Iron and Steel Co., Ltd., Japan.

Hirohata Works of Fuji Iron and Steel Co., Ltd. was faced with decreasing sintering rates due to loss of bed permeability as a result of using finer iron bearing materials while steel production was given to research and pilot plant work in the US and Sweden basic sintering burden is made up of 40 pct pyrite cinder, 40 pct fine ores and 20 pct mill scale and iron sands. Serious consideration was given to research and pilot plant work in the US. and Sweden covering pellets and a decision was made to pelletize the fines to about 3 to 5 mm diameter and mix them with other charge materials for sintering. The pyrite cinder from flotation and suspended bed roasting was difficult to pelletize, but additives such as bentonite, calcium hydrate and sticky ores have made success possible. Bed permeability and production has increased. In 1957, drum, cone, and disc pelletizers were used in a greenawalt pan plant of 60,000 L.T./month capacity. In 1960, evaluation of results governed the installation of four disc pelletizers in a new Dwight-Lloyd type plant, where they are operating satisfactorily. A report is made covering research and development work commenced in 1954 through the intermediate tests and final application to the present operation. Graphs and charts are included.

Relative Reduction Rates of Porous Iron Oxide Pellets:
C. L. Sollenberger and W. J. Helfrich, Allis-Chalmers Manufacturing Co.

The reduction of porous hematite and magnetite pellets was studied at 1850°F with pure reducing gases (H_2 and CO), and with various mixtures of reducing gases diluted with CO_2 and N_2. The effect of added fuel was also investigated. The reduction was followed with a thermogravimetric apparatus. With porous pellets an initial rapid reduction is obtained due to surface reduction of individual grains within the pellet. Diffusion of gases into the pores, which is dependent upon gas velocity, determines the extent of surface reduction in the interior of the pellet. Dilution of reducing gas with N_2 or CO_2 slows down the reduction markedly. This decrease in rate can be partially offset by the addition of solid fuel to the pellet. Final reduction of residual oxide particles appears to be rate controlled at the oxide-metal interface and takes place by topochemical processes. Absolute reduction rates do not appear to be applicable to porous iron oxide pellets.

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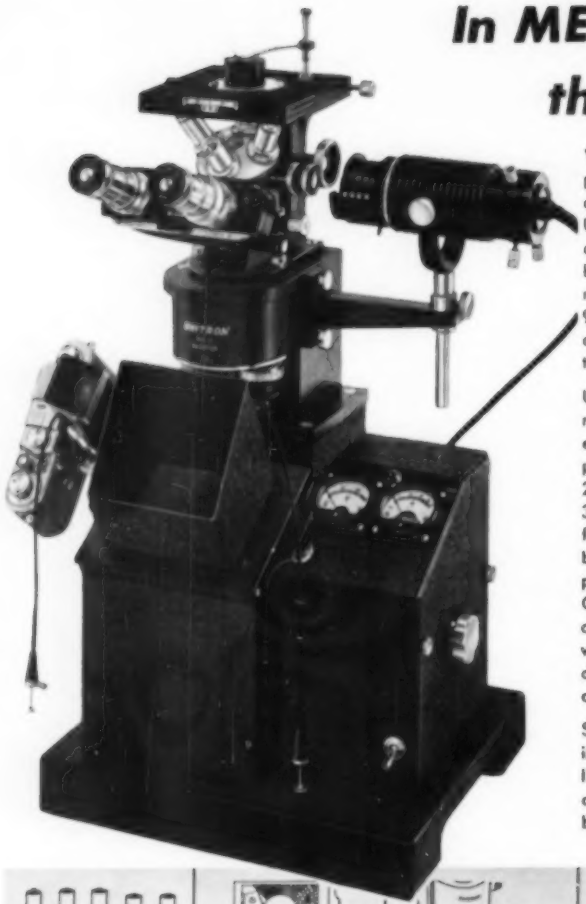

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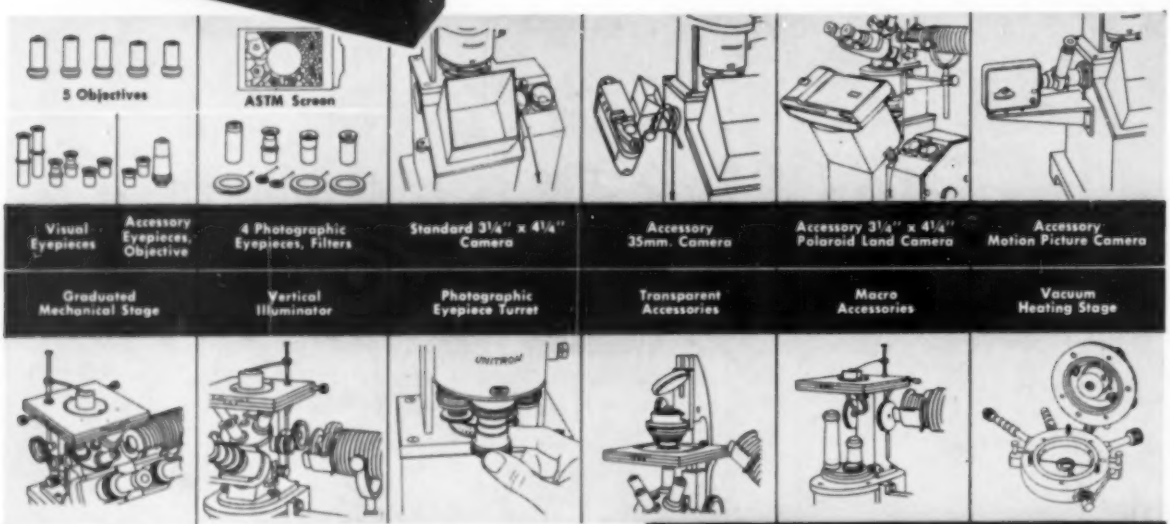

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